TECHNICAL AND OPERATING MANUAL:

TOXICS DEPOSITION

MONITORING PROGRAM

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TECHNICAL AND OPERATING MANUAL
TOXICS DEPOSITION MONITORING PROGRAM

Report prepared by:

M.N. Shackleton
Air Resources Branch
Ontario Ministry of the Environment

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1 INTRODUCTION

It is now well established that long range transport followed by deposition is a major; and may be the predominant pathway for the entry of toxic substances to the Great Lakes. Since 1987, polychlorinated biphenyls (PCB), selected chlorinated pesticides and trace metals have been monitored in the Ontario Ministry of the Environment Toxics[1] Deposition Network centred on the Great Lakes. More recently, the target list has been expanded to include dibenzodioxins and dibenzofurans (PCDD/PCDF) in precipitation and PAH in air samples and methods are being developed for many of the non-chlorinated pesticides.

This report documents the current status of instrumentation, sample handling and collection procedures, and the field data reporting and documentation requirements to monitor for all the target substances except trace metals. The instruments and sampling methods for trace metals are described in detail in the APIOS Deposition Monitoring Program Technical and Operating Manual , MOE Report # ARB-082-87-AQM (Bardswick, 1987) and are not reiterated here.

The manual has been prepared to guide 'field' personnel in carrying out their respective duties and responsibilities. It is not intended to replace adequate operator training in these procedures.

The specific objectives of these procedures are to ensure that:

- representative air and precipitation samples are collected,
- sample quality and integrity is maintained for chemical analysis,
- sampling procedures are consistent and reproducible at each site,
- sampler downtime is minimal and no samples are missed,
- all primary and ancillary field data are collected and documented.

Development of the sampling procedures was based on guides, journal articles, reviews, internal correspondence and the experience of network operatives. The procedures are designed to include specific quality control checks and are to be

[1]refers to all compounds monitored including trace metal as well as organics

supplemented with an adequate program of quality assurance. Network quality assurance and quality control are described fully in the Toxic Deposition Monitoring Program Quality Assurance Plan , Report No. ARB-060-90 (Ministry of the Environment, 1990).

Each section and chapter can be referred to independently. The text is designed so that revisions and improvements to sampling methodology may be readily incorporated into the body of the manual with a minimum of disturbance to the remaining text.

1.1 Explanation of Terms

Henceforth, the term 'field sample collection day' may be referred to as a 'changeover' day. Both expressions are appropriate. It is important to note that as expressed within this manual, the term 'changeover' embraces the collection of exposed (or 'active') samples followed by the installation of fresh 'clean' samples.

The term 'organics' is used throughout the manual as a generic expression for the entire complement of organic 'compounds' which are monitored in the network. Pesticides are chlorinated compounds which have herbicida¼õl or insecticidal properties. Individual compounds are not expressly referred to unless their identification requires specific procedures (e.g. PCDD/PCDF). The abbreviated expression 'RFA/Field sheet' is used throughout the manual in place of 'Request for Analysis and Field Information Sheet' (see chapter 7.0). The term 'jug sampling' expressly refers to the direct collection of unfiltered precipitation, a method being evaluated for the collection of PCDD/PCDF. 'Cartridge sampling' is the term used to describe the method whereby filtered precipitation is passed through an XAD-2 resin column '*in situ*' and the resin submitted for analysis. HiVol is used throughout the text as an abbreviated expression for 'high volume air sampler'. 'Site Operator' is the person actually performing sample changeover.

2 CHLORINATED COMPOUNDS IN PRECIPITATION

This chapter describes the equipment and procedures to sample for most organochlorines in precipitation. Note that sampling for PCDD/PCDF in precipitation is discussed in a separate chapter (Chapter 3). This is because the protocol to sample for PCDD/PCDF differs from that described in this chapter in several important areas, although it is similar in most respects.

2.1 Instrument Description

Precipitation inputs of organochlorine pesticides and PCBs are assessed over 28 days with automated M.I.C. Type B wet only collectors produced by MIC Co. in Thornhill, Ont. (Figure 2.1). The MIC collectors are equipped with a square Teflon-coated, stainless steel funnel to collect precipitation.

A stainless steel, Teflon coated hood completely covers the collection surface between precipitation events. The hood opens to expose the funnel during events. A Teflon-coated, polyurethane foam gasket attached to the underside of the hood forms a tight seal between the top of the collection vessel and the underside of the hood to prevent sample contamination, eliminate dry deposition collection between events and reduce sample evaporation.

A moisture sensor, consisting of 2 exposed faces tilted at 20° from the horizontal, is held by an arm slightly above and 0.5 m. to the side of the sampler. The sensitivity of the sensor grids is adjusted to 220 K ohm and the delay time is set at 1 minute 30 seconds. The sensing faces are slightly heated to ensure response during cold temperatures and snow-fall events.

The electronics and the motor are enclosed in a small compartment under the horizontal surface adjacent to the funnel. This surface is covered by a stainless steel screen designed to eliminate rain splash into the funnel. The motor turns a chain which rotates a shaft to which the hood arms are attached. An adjustable limit switch is set at the maximum rotation of this shaft to open a circuit and stop the hood movement when fully open. The motor compartment cover and splash screen are affixed to the sampler by 4 bolts which must be removed to gain access to the motor and electronics. An on/off toggle switch is located on the forward

vertical surface of the motor box.

The legs and chassis of the sampler are constructed of 0.3 cm cast aluminum. With the cover closed, sampler dimensions are: 1.5 m high x 1.0 m long x 0.5 m wide. The lower part of the sampler is enclosed by an insulated, stainless steel shelter. The shelter is equipped with a double-hinged door. During warm weather this door is replaced by a screen to reduce internal heating of the cartridge compartment.

2.1.1 Shelter Modifications

Minor modifications to the stock sampler are required for winter operation. Essentially, each sampler is insulated, sealed and heated to eliminate cartridge breakage due to winter freezing and minimize temperature variations inside the sampler. The funnels are individually heated during cold-weather sampling with thermostatically controlled thermofilm coated onto the undersurface. This ensures that snow-melt is completely collected and does not remain in the funnel for an appreciable length of time. The internal cartridge compartment is warmed by a thermostatically controlled heater (5-10 °C) during cold-weather sampling.

- Obtain the following materials:

1	4'x8' sheet of styrofoam
1	tube of clear silicone seal
5	2" squares of velcro
1	tube of Krazy glue
2	sheets of thermofilm
1	thermostat for thermofilm
24"	insulated wire (16-18 gauge)

To insulate the shelter:

- Remove the bolts attaching the motor compartment to the sampler housing. Lift the motor compartment off the housing and place it to one side. Take care that the power cord on the bottom of the motor compartment is not damaged by resting it on the ground.

- Glue 1" to 2" thick styrofoam onto the interior walls (including the

door) and floor of each MIC Type B shelter with silicone. Do not use contact cement or other glues as they may react with the styrofoam and cause sample contamination. Ensure that the styrofoam does not inhibit door movement.

To install the thermostat and heat the funnel collection surface:

- Remove the 4 screws holding the motor compartment cover in place. Remove the cover. Firmly attach a single pole, double throw thermostat onto the base of the MIC B motor compartment. Ensure that the thermostat placement does not interfere with opening and closing of the hood.

- Locate the thermostats temperature sensitive bulb outside the compartment in order to ensure thermostat response to external temperature changes. If necessary drill a hole to pass the bulb through the side of the compartment. Set the thermostat response temperature to 10°C.

- Remove the screws holding the funnel in place. Lift the funnel out and turn it over so that the funnel outlet is pointing up.

- Connect two sheets of thermofilm with a 6" length of 16-18 gauge shielded wire. Solder the wire to the thermofilm. Solder a wire lead to each sheet of thermofilm. Each wire lead must be long enough to reach the thermostat installed inside the motor compartment.

- Attach the connected sheets of thermofilm to the underside of the funnel with silicone. Secure the edges of the thermofilm heating pads to the underside of the funnel with duct tape.

- If necessary, drill a small hole in the base of the motor compartment to pass the wire lead from the thermofilm through to the thermostat.

To wire the thermofilm to the thermostat:

- Connect one lead directly from the thermofilm to the blue screw (open-on-rise) on the thermostat in the motor compartment. Connect the second thermofilm lead to the bridge where the internal heater

lead and white lead from the power cord connect in the motor compartment.

- Run a third wire to the red screw (common) on the thermostat from the bridge at the point where the motor fuse lead and black lead connect.

To install the Teflon hood gasket onto the underside of the hood:

- Glue the Velcro pads onto the underside of the instrument hood with Krazy glue to match the pads already attached to the Teflon gasket. These should secure the Teflon gasket in place except when the gasket is removed to be replaced.

2.2 Sampling Train Description

Dissolved and particulate species in rain/snow are collected and composited over multiple events. The chlorinated pesticides and PCB are collected on an *'in situ'* gravity fed XAD-2 resin adsorbent column. A glass-fibre particulate filter is located upstream of the resin column. Trapping of the organic content on the resin helps to minimize losses by volatilization and also provides a "preconcentration" step for analysis.

The individual components of the sampling train are described proceeding in the direction of flow. Illustration of the cartridge and filter set-up are provided in Figures 2.2 and 2.3.

2.2.1 Collection Funnel

The funnel is 0.46 m on a side with 0.30 m deep, vertical sides. The collection surface area is about 0.21 m^2. The funnel slopes about 20° from the base of each side towards a 2 cm wide, circular drain. The relatively large collection surface permits the collection of the large precipitation volumes (greater than 10 l) needed to detect the low concentrations of organics present in precipitation. The collection surface of the funnel is coated with Teflon (0.076 cm) to minimize contamination and adsorption by fine particulate matter to the collection surface.

2.2.2 Funnel Adaptor Components

The outlet of the collection funnel is internally threaded to receive the Teflon 6" extender tube. The Teflon extender is custom machined from 1 3/8" extruded Teflon rod and is used to increase headspace, improve flow and act as a reservoir to keep the particulate filter moist. Moulded rod is not used because of greater difficulty in machining it.

The bottom of the extender is externally threaded to fit into the Teflon funnel adaptor. The Teflon adaptor is custom machined from extruded Teflon rod 1 5/8" in diameter. One end of a male valve adaptor threads into the funnel adaptor. The other end of the male valve adaptor threads into a Teflon flow cut-off valve (flow control spigot). The flow control spigot is used to stop passage of the precipitation through the sampling train and permit filter replacement even when the collection funnel is full.

2.2.3 Particulate Filter

The Nacom filter pack threads into the bottom of the flow control spigot (1/4" NPT). The particulate filter is a Gelman Sciences Type A-E 47 mm glass fibre filter which is soxhlet washed to ensure that the filter is not contaminated with organics. To improve particulate distribution over the filter surface and reduce clogging, the filter pack fitting above the filter disc is machined out to provide greater headspace. A Teflon column adaptor connects the filter pack with the glass collection column.

2.2.4 Collection Column

The collection column is borosilicate glass with a 300 mm effective length, and a 15 mm internal diameter with internally threaded ends. A Teflon bottom drip adaptor with a flow regulator valve is screwed into the downstream end of the column. The flow-regulator valve is completely closed during shipping or handling and completely open during sampling.

The adsorbent surface consists of approximately 25 g (70 mm bed) of soxhlet washed XAD-2 resin beads, stored in methanol. The resin is added to the column at the laboratory as a methanol slurry. Glass-wool plugs are located upstream of the resin bed to remove particulate matter and to hold the resin in place. A screen in the bottom drip-adaptor prevents any loss of the resin beads through the downstream end of the column.

2.2.5 Flow Control

The precipitation effluent feeds from the collection column through Teflon tubing which leads from the bottom drip adaptor, up through a loop above the filter pack at the base of the collection funnel and ends at the mouth of the large effluent jug, at a level slightly below the filter pack. The Teflon tubing has an internal diameter of 1/16", with 0.03" thick walls and is about 4' in length. The tubing is looped above the filter pack to create a hydraulic head keep the filter moist and eliminate bubbles developing in the fittings at the upper end of the sampling train.

2.3 Changeover

This section describes the procedures to collect exposed cartridges and filters and replace them with fresh cartridges and filters. The term 'changeover' covers both the collection of active samples as well as the installation of fresh 'clean' samples. Changeover occurs only at pre-scheduled 28-day intervals and always falls on a Tuesday. Changeover should be started on each scheduled Tuesday as early as possible, preferably at 8am.

Handling of filter packs as well as the entire organics in precipitation changeover procedure should be performed with clean, bare hands or while wearing cotton gloves. *Under no circumstances should changeover be performed while wearing plastic gloves.*

2.3.1 Preparing for Changeover

In preparation for changeover the shipping technician will pick up all precipitation cartridges from the laboratory and ship them to each site at least one week prior

to changeover. After shipping the fresh samples the shipping technician will contact each site operator no later than the Friday before changeover to:

- confirm that the cartridges and/or jugs have arrived in good condition,
- ensure that the site operator has sufficient supplies, materials and equipment on hand to perform sample changeover,
- ensure that the site operators have adequate filters on hand to re-load filter packs for changeover and replace filters in the field when clogged.

To prepare for Organics in precipitation changeover the site operator must prepare all supplies and materials required for the organics-in-precipitation sample changeover and to perform any maintenance required (see chapter 5), and clean and load filter pack(s) (see sections 2.3.1.1, 2.3.1.2 and 2.3.1.3). If supplies are needed, the site operator must contact the shipping technician in advance for replacements to be delivered in time for changeover.

The list of materials required to collect an exposed cartridge and filter and replace them with fresh samples are:

- 3 clean Teflon squeeze bottles
- 3 full Winchesters (methanol, acetone, distilled/deionized H_20)
- 1 Clean PCB bottle to collect final rinse
- 1000 ml graduated cylinder
- glass funnel
- Hand pump
- Kimwipes
- aluminum foil
- small brush (to clean funnel opening)
- cotton gloves

2.3.1.1 Unloading Filter Packs

- Lay clean sheet of aluminum foil on clean working surface.
- Grasp filter pack with the side marked 'IN' facing up. Twist

grey locking ring to open the active filter pack. Lift upper portion of filter pack off and place to one side.

- Remove the filter from lower portion of filter pack with Teflon-coated tweezers and place the filter on the clean sheet of aluminum foil. Ensure the dirty side of the filter faces up.
- Fold the aluminum foil in half so that the crease crosses under the middle of the filter and the filter doubles over on itself inside the aluminum foil. The dirty side of the filter must be on the inside of the folded filter. Fold the foil envelope edges to seal the filter within and attach a blank label to the aluminum foil filter packet.
- Write on the label the **'Filter Type'** (i.e. Active Filter or Handling Blank Filter), **Date of Collection** and **Site Name.**
- Place the packet containing the filter in a clean whirl-pak and seal.

2.3.1.2 Cleaning Filter Packs

- Separate the dirty filter pack components. Wash and scrub the filter pack parts with soap (Alconox) and water. Remove all visible dirt. Rinse each filter pack part with methanol. Repeat the rinse with distilled water, place on a clean Kimwipe and let air-dry.
- When replacing a filter in the field rinse and scrub each filter pack part only with methanol. The filter pack may be re-loaded while still wet when changing a filter in the field.

2.3.1.3 Loading Filter Packs

- Separate a clean filter pack. Hold the inlet part of the filter pack with the machine-out cone facing up.
- Grasp a fresh filter with Teflon-coated tweezers. Place the filter on the inlet side of the filter pack adjacent to the machined-out cone.
- Re-attach the downstream half of the filter pack. Do not disturb the filter placement. Tighten the grey locking ring

around the filter pack securely.

- If the filter pack is not to be used immediately wrap the filter pack in clean aluminum foil and store for later use.

2.3.2 Instrument Operation

This section describes how to briefly check instrument operation prior to removing an exposed sample and/or installing a new sample. To check instrument operation after arriving at the site:

- Touch the sensor grid with a moistened finger to activate the hood and expose the collection funnel. The hood movement should be relatively smooth with only a small amount of play after the hood begins to drop into the open position. If the hood does not open, identify and rectify the problem before proceeding with the sample collection procedure (See section 5.2.2 on troubleshooting mechanical problems). If it is raining or snowing, the hood should be in the open position, or may oscillate back and forth.

- While the hood is open, check for precipitation pooling in the collection funnel. If precipitation has pooled in the collection funnel it may be necessary to adjust the sampling train to increase flow and drain the pooled precipitation more rapidly (See section 5.2.3 on troubleshooting sampling train problems).

- Wait until all of the precipitation drains from the collection funnel before proceeding to collect the sample cartridge and filter pack. If precipitation has not pooled in the collection funnel simply wait until the hood returns to the closed position then turn the instrument off before collecting the exposed samples.

- Record the response time in the field note book needed for the hood to return to a resting position. The hood should return to the closed position about 1 min. 30 sec. after touching with a moist finger, or at the end of a precipitation event. If it is raining or snowing the hood may already be open and the response time cannot be checked. If so, simply proceed with the sample collection procedure

2.3.3 Sample Collection

This section describes the protocol for removing cartridges and filters that have been exposed, for measuring effluent volume and for collecting a final rinse of the collection surface.

2.3.3.1 Active Cartridge

To remove the active or exposed cartridge:

- Shut off flow from the cartridge to the effluent jug by closing the drip control knob at the bottom of the active cartridge. Do not over-tighten. Do not permit any water to drain from the cartridge itself.
- Unscrew the spaghetti tubing from the base of the resin cartridge. Take care that the other end of the tubing remains in the mouth of the effluent bottle. Drain all excess effluent from the tubing into the effluent bottle. Roll the used tubing up and place the tubing to one side in a plastic bag. Return the used tubing to Toronto with the active samples.
- Lower the height of the metal jack which supports the effluent jug and remove the jug from inside the sampler without spilling.
- Unscrew the resin cartridge from the top cartridge adaptor fitting and replace the top cartridge adaptor fitting on the cartridge with a clean Teflon plug and 'O' ring. The Teflon plug and 'O' ring originally taken from the cartridge may be re-used if they were wrapped in foil and stored in a clean, dry place inside the housing during the sampling period.
- Attach a blank sample label directly to the cartridge. Write clearly on the label with a pencil or china marker. Do not use a pen as the ink may run if wet. Label the sample **'active cartridge'** and list the **date of collection** and **site name**.
- Wrap the resin cartridge in protective plastic padding (bubble wrap)

and store it in the aluminum tube provided for cartridge storage and transport.

- Remove the top cartridge adaptor fitting from the outlet of the filter pack and place it on clean aluminum foil on a flat surface. Keep it for later use. Do not lose the 'O' ring attached to the fitting.

2.3.3.2 Active Filter Pack

To collect the active filter pack:

- Unscrew the active filter pack from the flow-control spigot and wrap the filter pack in fresh aluminum foil. Remove the active filter from the filter pack later (except when a fresh pre-loaded filter pack is not available).

- Attach a blank sample label to the foil packet. Identify the sample on the label i.e. **'Active Filter Pack'**. Include the **date of collection** and the **site name** on the label.

2.3.3.3 Effluent Volume

To measure effluent volume:

- Transfer the effluent from the effluent jug into a 1000 ml graduated cylinder with a hand pump. Fill, empty and refill the cylinder as many times as required until all of the effluent has been measured. Record this volume. Discard the effluent.

- If desired site operators may measure and mark volume in one litre increments on the side of each effluent jug. Indelible ink must be used. If the jugs are accurately marked it is only necessary to transfer the portion of effluent lying between two one litre increments to a graduated cylinder for measurement.

- Calculate the effluent volume by adding the volume of water in the

jug as measured by the nearest marked 1 litre increment to that transferred to the graduated cylinder.

2.3.3.4 Final Rinse

After the exposed filter pack and cartridge are removed the funnel surface must be rinsed with acetone and the rinse collected. Acetone funnel rinses are submitted as container #2 for the cartridge and are given the same sample submission number as the cartridge. Funnel rinses collected using other solvents are submitted separately and are given unique submission numbers.

You will be informed if a solvent other than acetone is to be used for the final rinse. If another type of solvent is used you will ensure that the sample is labelled accordingly and the solvent type noted on the field sheet.

To collect a final rinse of the funnel surface:

- Ensure the sampler is turned off and manually move the hood to the open position to expose the collection funnel.
- Close the flow-control spigot.
- Wipe a glass funnel with Kimwipes soaked in acetone. Rinse the interior and exterior glass funnel surfaces with acetone. Rinse a graduated cylinder with a small amount (50 ml) of acetone. Swirl the acetone around the graduated cylinder to rinse the interior walls. Discard waste rinse in waste bottle.
- Stand the funnel in the mouth of a clean PCB bottle. Place the PCB bottle and funnel directly under the flow-control spigot outlet. Use the jack to raise the PCB bottle and funnel up to eliminate splashing.
- Measure *exactly* 250 ml of acetone in the clean, rinsed graduated cylinder. Carefully, pour the acetone into the collection funnel. Make sure that the sides of the collection funnel are thoroughly rinsed.
- Slowly, open the flow-control spigot making sure that all of the solvent drains into the funnel and is collected in the PCB bottle.

Label the bottle '**(Acetone) Final Rinse**' and show the **site name** and **date of collection** clearly. Store with the cartridge and filter pack for laboratory submission.

2.3.4 Sample Installation

This section describes the procedures to clean the sampler prior to installing a new 'unexposed' cartridge and filter and the protocols for installing the unexposed cartridge and filter. Note that the filter pack and cartridge are installed in reverse order from their collection.

2.3.4.1 Instrument Cleaning

The instrument must be cleaned before a fresh cartridge and filter are installed. After the cartridge and filter pack have been removed and a final rinse collected, and while the hood is in the open position:

- Wipe the underside of the hood with acetone-soaked kimwipes.
- Place a small pail or waste bottle under the flow-control spigot beneath the collection funnel inside the housing unit. Use a soft bristle brush and acetone to scour the inside of the funnel drain hole, the Teflon funnel adaptor and the entire funnel surface.
- Wipe the entire collection surface and the inside of the funnel outlet with acetone-soaked kimwipes to remove any residue. Continue until surface dirt is no longer being removed from the collection surface (i.e. dirt is not being transferred to clean kimwipes by scrubbing).
- Rinse all exposed surfaces first with acetone and then twice with distilled/deionized water into the waste pail or bottle.
- Place all used Kimwipes in a sealable plastic bag for later disposal.
- Manually, close the hood.

2.3.4.2 Fresh Filter Pack

To install a new filter pack:

- Unwrap a fresh, loaded filter pack.
- If a clean freshly loaded filter pack is not available the '**active filter pack**' must be unloaded, cleaned, re-loaded with a fresh filter while at the site and re-used in the field sampler. Follow the procedure for unloading, cleaning and loading filter packs described in sections 2.3.1.1, 2.3.1.2 and 2.3.1.3.
- Saturate the filter in the filter pack by spraying distilled water through the inlet of the filter pack. Do not poke the nozzle of the Teflon spray bottle in the inlet. This may damage the surface of the filter.
- Wrap Teflon tape around the inlet of the filter pack and thread the filter pack into the outlet of the cleaned flow-control spigot with the side marked 'IN' facing up. The filter pack is now in position to permit the passage of precipitation.

2.3.4.3 Fresh Resin Cartridge

To install a new cartridge:

- Wrap Teflon tape around the outlet of the filter pack.
- Prepare a clean cartridge adaptor to replace the end plug in a fresh cartridge. The cartridge adaptor taken earlier from the active cartridge may be re-used if rinsed with acetone first. Ensure that the cartridge adaptor is equipped with an 'O' ring.
- Remove the top end plug from an unused, prepared collection cartridge and thread the cartridge adaptor into the open end of the collection column.

- Wrap the end plug in aluminum foil if a handling blank is not required and store it in the instrument housing.

- Thread the cartridge adaptor with cartridge attached onto the bottom of the filter pack.

- Screw fresh Teflon tubing with ferrule and drip-control valve adaptor into the bottom of the collection cartridge drip-control valve. Run the tubing outside the sampler.

2.3.4.4 Activating the Fresh Resin Cartridge

The XAD-2 resin is loaded at the laboratory into the cartridge as a methanol slurry. The methanol in the cartridge must be displaced by distilled/deionized water before being left to collect a sample. It is also necessary to check that flow through the sampling train is smooth and unimpeded before leaving the site. Troubleshooting sampling train problems are described in section 5.2.3.

To activate the fresh resin cartridge:

- Manually move the hood to the open position. Turn the sampler off once the collection surface is exposed. Close the flow-control spigot.

- Measure approximately 250 ml of distilled/deionized water and pour into the funnel. Let the water partially drain from the funnel. This flushes the methanol out of the collection cartridge. When the water surface reaches the opening to the funnel neck loop the tubing above the filter pack and secure it with a cable tie to the funnel adaptor.

- Place a clean effluent jug on the metal stand inside the sampler housing. Raise the jug height until the mouth of the jug is higher than the free end of the tubing. Secure the free end of the tubing leading from the cartridge in the effluent jug mouth.

- Once the dripping has ceased, inspect the inside of the funnel opening to ensure that a small reservoir of standing water completely covers the filter. The surface of this reservoir should be within 2" of the

funnel opening in the base of the funnel.

- Add small amounts of water and adjust the tubing until the reservoir level is satisfactory. Dislodge any bubbles that have formed above the filter pack using a clean cable tie with the sharp point removed. Gently lower the cable tie into the sampling train until the blunted end lightly rests on the filter. Carefully, agitate the water with the cable tie to release any precipitation bubbles. Precipitation bubbles in the collection cartridge should be dislodged by lightly tapping the side of the collection cartridge.

- Seal the mouth of the effluent jug with fresh aluminum foil and turn the sampler back on and briefly check to ensure hood movement and instrument operation.

2.3.5 Handling Blank

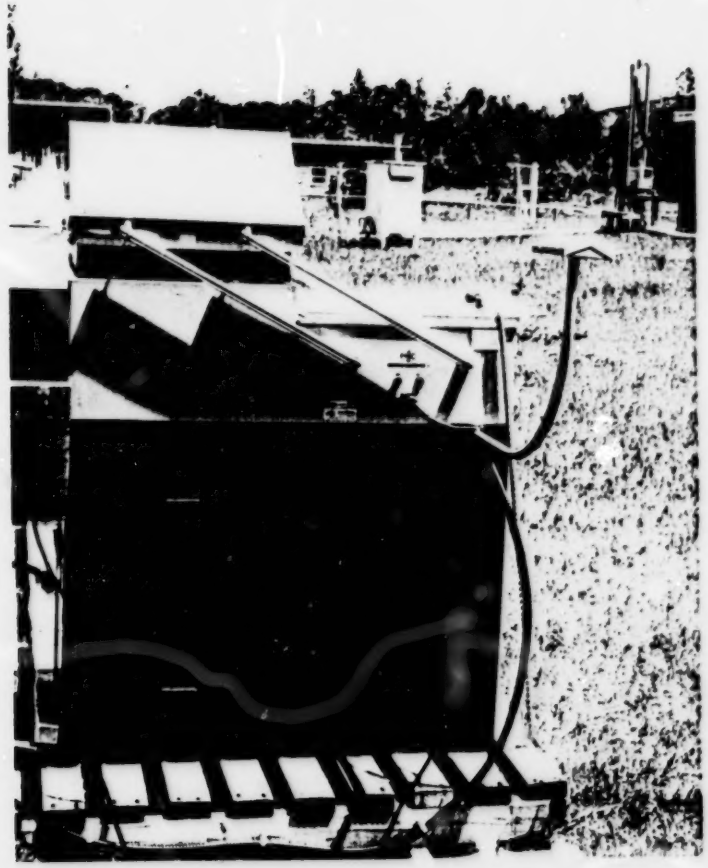
The procedure for collecting a handling blank is exactly the same as that described in sections 2.3.4.2, 2.3.4.3, and 2.3.4.4 except that the handling blank is not left in place to collect a precipitation sample but is removed immediately after activation and stored inside the sampler.

- Once the last of the distilled/deionized water has drained out of the collection funnel to the top of the handling blank cartridge, close the drip-control valve at the bottom of the cartridge. Do not permit any water to drain from the cartridge itself.

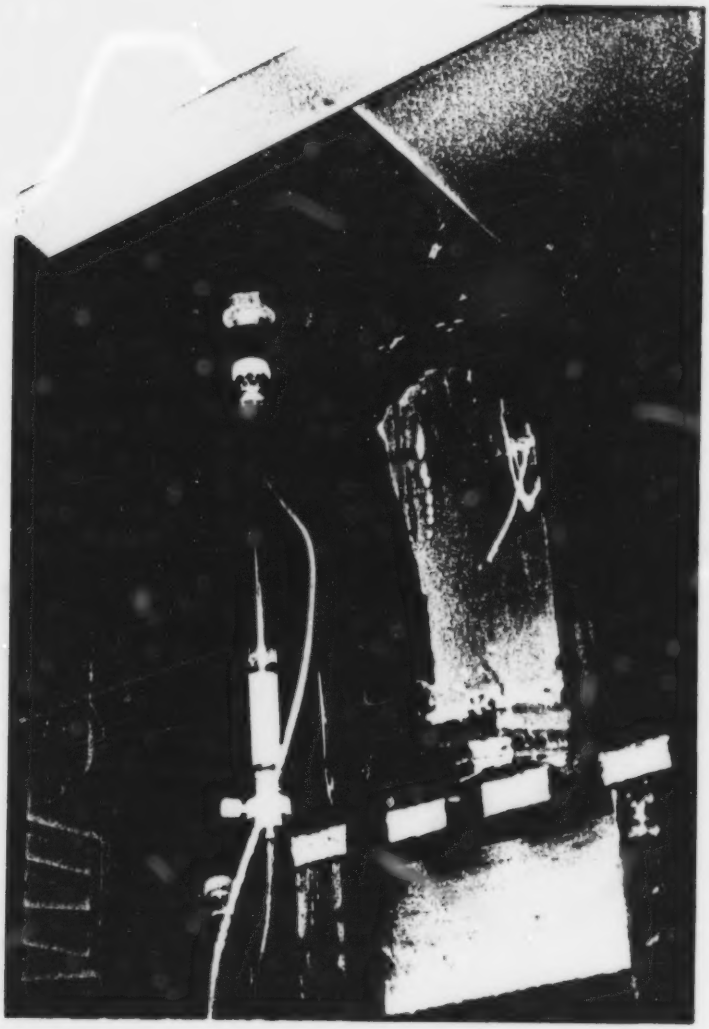
- Unscrew the handling blank cartridge from the top cartridge adaptor fitting. Thread the clean Teflon plug removed earlier from the cartridge back onto the cartridge in place of the adaptor. Place the cartridge in the aluminum tube provided, and label the tube **'handling blank'**. Label the tube with the **site name** and **date of collection** also. Store the **'handling blank'** in the tube inside the MIC housing until the next changeover date.

- Unscrew the filter pack from the funnel adaptor and wrap it in aluminum foil for future disassembly. Label the filter pack **'handling blank'** and denote the **site name** and the **date of collection.**

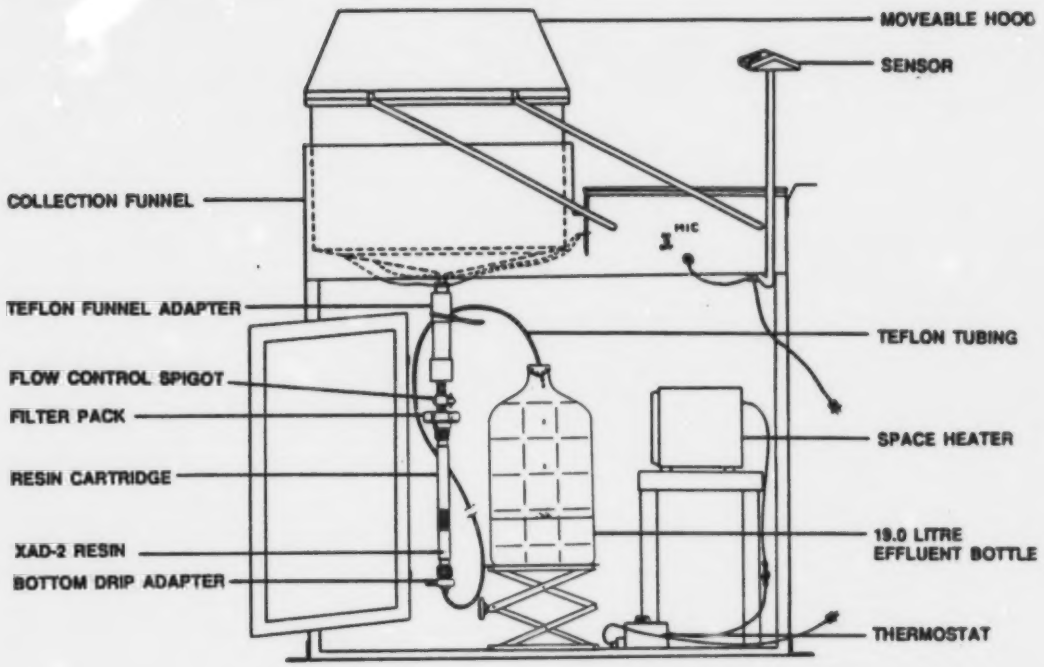
2.1: Photograph of an MIC Type B Precipitation Collector



2.2: Photograph of the Interior of an MIC Type B Precipitation Collector and Sampling Train



2.3: Diagram of an MIC Type B Precipitation Collector and Cartridge Sampling Train



3 PCDD/PCDF IN PRECIPITATION

Chapter 3 focuses on the specific protocols used for PCDD/PCDF sampling and should be read in light of the more extensive discussion in chapter 2. It is important to note that the protocols for sampling of PCDD/PCDF in precipitation parallel those used for the collection of chlorinated organics (Chapter 2). In the absence of specific instructions to the contrary assume that methodologies are the same as described in chapter 2. The order of discussion is the same as in chapter three; sample collection is first followed by sample installation.

3.1 Instrument Description

Precipitation inputs of PCDD/PCDF are assessed over 28 days with automated M.I.C. Type B wet only collectors produced by MIC Co. in Thornhill, Ont. (Figure 2.1). The principal features of this instrument are the same as the MIC Type B samplers used to sample for chlorinated pesticides and PCB (Chapter 2) i.e.

- sensor grids extend out from the body of the collector to detect precipitation.
- The hood is automated to open only during precipitation events. The moisture closes an electronic circuit on the sensor surface which activates the hood mechanism.

3.2 Sampling Train Description

Two alternative sampling protocols for PCDD/PCDF are being evaluated. In the first protocol, unfiltered precipitation is collected in a large glass jug and submitted for analysis (Figure 3.1). The second protocol uses an XAD-2 resin cartridge similar to the one described for chlorinated pesticides and PCB in chapter 2.

Prior to sampling in the field the glass jugs are prepared in the laboratory by spiking a solution of distilled/deionized water with known concentrations of $^{13}C_{12}$ PCDD. The XAD-2/methanol slurry in the resin cartridges is similarly spiked. The recovery of the spiked $^{13}C_{12}$-labelled PCDD in the cartridges and jugs is used

to calibrate the overall sample recovery.

The sampling train used for jug sampling is quite different from that used for cartridge sampling. Unlike the chlorinated organic or PCDD/PCDF cartridge sampling trains, the jug collection method does not employ an upstream particulate filter. Precipitation flows directly, without being filtered, from the funnel into the glass sample container. The collected precipitation is stored in the jug for up to 28 days.

The collection jug rests on a metal jack. The jack is used to raise and lower the jug for changeover and to ensure that the mouth of the spiked sample jug fits over the neck of the funnel when sampling. The connection between the jug and the funnel is tightly sealed with aluminum foil to minimize external contamination. A Teflon collection funnel adaptor is used to ensure the passage of precipitation into the jug.

3.3 Changeover

The procedures for collecting PCDD/PCDF cartridges are the same as those described to collect organics cartridges (Chapter 2). These procedures are not reiterated here. The only exceptions are that acetone is replaced with methylene chloride as the solvent of choice, the final rinse volume is 500 ml rather than 250 ml as used in the organics changeover and effluent volume is not determined in the field.

The procedure used when using the jug collection method to sample for PCDD/PCDF is described below:

3.3.1 Preparing for Changeover

The preparations required for PCDD/PCDF changeover are essentially the same as those required for chlorinated compounds in general. There are some changes to the list of required materials and procedures when sampling for PCDD/PCDF in precipitation. The differences are summarized below.

- Acetone is not required in the PCDD/PCDF changeover procedure.

- The solvent that replaces acetone is methylene chloride. Note that methylene chloride has different safety and handling requirements than acetone.

- A replacement filter pack and spare effluent jug are only required when the cartridge protocol is being used for PCDD/PCDF in precipitation monitoring. Collected precipitation is not filtered when the aqueous jug collection method is used.

- The procedure to load, clean and unload filter packs is the same as described in chapter 2 when the cartridge procedure is used.

- Handling blanks are not rotated among sites but are collected at every site each changeover.

3.3.2 Sample Collection

This section describes the procedures to collect the exposed 'field blank' and 'active' jug samples, and to collect a final rinse sample of the collection surface.

3.3.2.1 Field Blank Jug

To collect the 'field blank jug':

- Open the sampler hood and check instrument operation as in Chapter 2. No precipitation should have collected in the funnel because the large drain at the base of the funnel is not impeded with a filter.

- Remove the jug containing the $^{13}C_{12}$-labelled PCDD field blank. Pack the field blank PCDD/PCDF-in-precipitation sample the same way as the active sample in a plastic carrying case.

3.3.2.2 Active Jug

To collect the exposed sample jug:

- Carefully, remove the aluminum foil used to seal the mouth of the 'active' collection jug and the funnel drain inside the MIC sampler.

- Lower the metal jack on which the jug sits. Remove the 'active' effluent jug from the instrument housing taking care not to spill or come into personal contact with the liquid. Although the concentration is low, the effluent is spiked with PCDD. Seal the mouth of the active PCDD/PCDF-in-precipitation bottle tightly with aluminum foil. Seat each bottle securely in its plastic carrying case.

3.3.2.3 Final Rinse

As in chapter 2, a final rinse of the funnel surface is performed:

- Repeat the steps for the collection of an 'organics final rinse' as described above, but substitute 500 ml of methylene chloride for acetone.

3.3.3 Sample Installation

This section describes how to install a fresh jug and prepare the instrument to collect a new sample.

3.3.3.1 Instrument Cleaning

- Clean the funnel, funnel opening and hood gasket with methylene chloride. The procedure is described in Chapter 2.

3.3.3.2 Fresh Active Jug

The fresh unexposed jug is installed next:

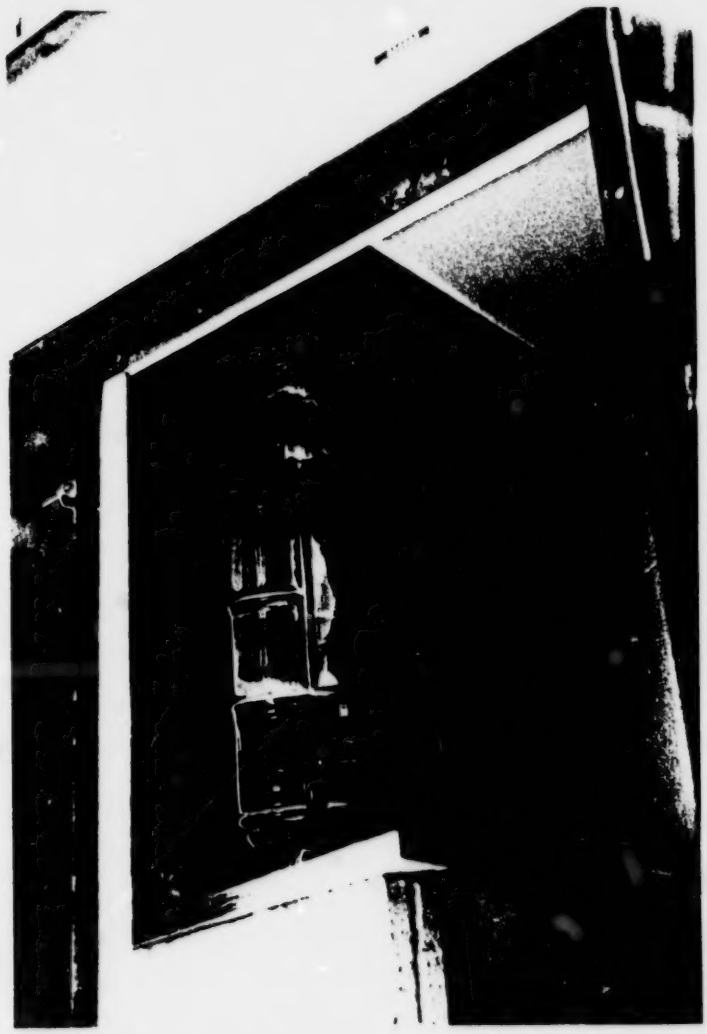
- Place the new 'spiked active' jug on the jack directly underneath the funnel opening and carefully remove the aluminum foil sealing the jugs mouth. Store the aluminum foil in a plastic bag to be discarded as a hazardous waste.

- Raise the jack until the mouth of the jug is completely surrounding but not touching the funnel opening. Seal this area with clean aluminum foil.

3.3.3.3 Fresh Field Blank Jug

- Place the field blank jug inside the housing, adjacent to the active sample until the next changeover date.

3.1: Photograph of a PCDD/PCDF in Precipitation Jug Sampling Train.



4 CHLORINATED COMPOUNDS IN AIR

The equipment and sampling protocols pertinent to sampling for organochlorines in air are outlined in this chapter.

4.1 Instrument Description

Andersen HiVol air sampling shelters, modified to accommodate glass fibre filters and XAD-2 resin cartridges, are used to monitor organochlorine pesticides and PCB in air. External dimensions of each shelter are 15"x14"x52". Each sampling shelter is fitted with a Lamb tangential discharge by-pass vacuum motor (Ametek Inc.). A mechanical timer and mass flow controller are used to control motor speed and set the sampling time.

4.1.1 Shelter Modifications

Modifications to the stock sampler are required for winter operation and to fit the sampling train inside. Essentially, each shelter is heated during the winter, sealed from the elements and extensively insulated. The following materials are required to modify each shelter:

3	0.08" thick anodized aluminum snow shields
1	0.08" thick extra base plate
1	1" or 2" thick sheet of styrofoam insulation
1	tube of silicone seal
1	tube of krazy glue
24"	insulated wire (16-18 gauge)
1	ceramic heater

To insulate and heat the shelter:

- Fit additional anodized aluminum plates around the base of the shelter to completely encase the motor and protect the sampling train from rain and snow. The snow shields may be removed during warm weather operation so that heat from the motor can dissipate.

Add an additional base plate to the sampler at ground level to complete the encasement of the motor compartment.

- Insulate the shelter with styrofoam insulation to minimize temperature variations inside the shelter. The insulation in the lower compartment should be removable for summer operation. The insulation in the upper compartment should be permanent. Seal all holes and openings in the housing with clear silicone.

- Bolt the ceramic heater in place in the upper compartment so that the site operator can not readily touch it and burn himself during changeover or repair.

To create sufficient space inside the shelter for the sampling components:

- Move the aluminum motor flange support plate upwards. Cut a 5" hole in the centre of the support plate and drill 3 holes corresponding to the positions of the motor mounting lugs.

- Cut two 2 1/2" holes in the back of the sampler to pass the exhaust hose through. The exhaust hose should be at least 50 ft. long.

- Cut a 2 1/2" hole near the right front corner of the support plate to pass the wires used to connect the flow controller, timer and motor together.

4.2 Sampling Train Description

Positioning of the sampling train inside the sampler is shown in Figure 4.1. All sampling components are constructed from aluminum and stainless steel to minimize sample contamination. The organics-in-air sampling components are illustrated in detail in Figure 4.2 and are described in sections 4.2.1 to 4.2.6, starting from the top of the sampling train and working downwards. Silicone gaskets or 'O' rings are used to seal all connections between sampling components. A complete list of silicone spacers and 'O' rings is included below and their positions in the sampling train illustrated in Figure 4.2.

- calibration orifice 'O' ring (size 338)
- internal cartridge and spacing ring 'O' ring (size 236)
- outer cartridge holder 'O' ring (size 236)
- flow controller sensor 'O' ring
- motor flange 'O' ring (size 238)
- motor 'O' ring (size 354)
- funnel horn gasket 1/8"x3"I.D.x4"O.D.

4.2.1 Particulate Filter

Suspended particulate matter is collected on an 8" X 10" Teflon-coated glass fibre filter (Pallflex) which has been subjected to the clean-up procedure. The filter is tightly held in place over the opening to the sampling train by a custom-made cast aluminum filter holder attached to the filter horn by butterfly nuts at each corner (Figure 4.3).

4.2.2 Resin Cartridge

Small particles and more volatile compounds in the vapour phase are collected on 45 g of XAD-2 (Amberlite) adsorbent resin in the stainless steel cartridge downstream of the filter. The resin cartridge rests on an aluminum ring inside the outer cartridge holder (Figure 4.2). The aluminum ring is equal in length to the resin cartridge and is used to position the resin cartridge about 1/8" above the rim of the outer cartridge holder.

The outer cartridge holder threads into a ring loosely fitted around the neck of the filter horn. Tightening the ring around the funnel horn causes downward pressure on the inner resin cartridge. This acts to seat the cartridge securely between the upstream (filter horn/cartridge holder) silicone spacer and a downstream silicone 'O' ring and helps prevent air leaks or air passing around the inner cartridge.

The aluminum ring may be removed and replaced by a second resin cartridge if necessary (i.e. for breakthrough validation studies or to contain a PUF slice to prevent organic re-entrainment from the motor to the sample cartridge).

4.2.3 Extension Column

An 8 in. long aluminum extension tube threads into the loose ring at the base of the cartridge holder. The internal diameter of the extension tube decreases from 3 1/4" to 2" in an inverted cone over a length of 2". The internal diameter remains 2" for the remainder of the extension tubes length (6"). The extension is used to station the flow controller probe away from the cartridge and stabilize the air-flow passing across the sensor. Experimentation has shown that a 2" diameter is the optimum size to maximize air flow while minimizing air turbulence, given the physical parameters of this sampling system.

The mass flow controller sensor probe is inserted into the centre of the extension tube air passage about 1" to 1 1/2" above the base. The flow controller probe is located downstream of the resin cartridge to reduce potential contamination from the probe and to accommodate any pressure drop across the cartridge. A hose clamp is used to seat the probe securely in place. Air leaks around the base of the probe are prevented by a small silicone 'O' ring.

4.2.4 Motor

A Lamb HiVol vacuum motor draws air through the sampling train. Minimum power requirements are 19 amps starting and 6-12 amps continuous operation (80-115 V AC). The motor samples approximately 0.68 m^3 per minute at maximum line voltage (approximately 115 v AC) with one cartridge containing 45 g of XAD-2 resin downstream of a Teflon glass-fibre filter. This is well above the desired flow-rate of 0.42 m^3 per minute. At 0.42 m^3 per minute the voltage to the motor is approximately 80 volts.

The motor was not designed to fit inside the shelter and must be installed correctly. The procedure to mount the motor inside the sampling shelter is described in section 4.2.4.1.

4.2.4.1 Motor Mounting

Once the shelter has been insulated and modified the motor may be mounted on the motor flange and installed inside the shelter. The following materials are

required to mount the motor in the shelter:

1	'O' ring for motor (size 354)
3	1 5/8" long motor mounting spacers
3	1/4" stainless steel bolts (with nuts and washers)
2	x50' exhaust hose and hose clamps
1	double-ported exhaust shroud fitting for motor
1	aluminum motor flange

To mount the motor on the motor flange:

- Replace the factory supplied motor cover with a custom-made, stainless steel, twin-exhausted motor shroud. Secure the replacement shroud in place by tightening the hose clamps. The motor shroud is readily removed for normal maintenance of the motor (i.e. brush replacement).

- Place an 'O' ring between the motor fan housing and the motor flange to prevent re-circulation of the motor working air and ventilating air. The 'O' ring should fit snugly in the circular groove on the upper surface of the motor.

- Slide 1x5/8" metal spacing tubes over 3/8" stovebolts between the motor mounting arms and the motor flange to ensure an even distribution of pressure on the motor gasket when bolted together.

- Without disturbing the motor 'O' ring (gasket), bolt the motor to the bottom of the custom-made cast aluminum motor flange with 3/8" stovebolts.

To install the motor and motor flange inside the shelter:

- Bolt the motor, gasket and motor flange securely onto the underside of the support plate.

- Outfit the two motor exhaust ports with a minimum of 50' of 2" I.D. exhaust hose. The exhaust hose outlet is always located away from the sampler and downwind of the prevailing wind.

4.2.5 Flow Controller

Moore and Gentry model 430/450 flow controllers or Levitt-Safety model WSAG 313 flow controllers with built-in mechanical timer and elapsed time indicator are used to regulate voltage to the motor. Both models work by compensating for clogging in the filter or resin cartridge by increasing the voltage supplied to the motor.

4.2.5.1 Flow Controller Mounting

The flow controller must be mounted inside the sampling shelter. Mounting the flow controller is described in this section. To install the flow controller inside the shelter:

- Drill 4 holes in the upper half of the Hi Vol door. These holes must match the mounting holes on the flow controller.

- Using the drilled holes, mount the flow controller on the inside of the Hi Vol door with the flow controller facing inward. This will expose the flow controller controls when the door is opened and permit easy maintenance. Check to ensure that flow controller placement does not interfere with opening or closing the door.

If a Moore and Gentry flow controller is being used, the internal 24 hour clock in the Moore and Gentry model 430/450 must be disconnected and replaced with an external 7 day mechanical timer before using.

4.2.6 Timer

Sampling start and finish is controlled by a multiple-event, 7-day mechanical timer. The timer may be external to the mass flow controller (Moore and Gentry) or may be incorporated within the same casing (Levitt-Safety). The timer can be set to sample automatically at any time within a seven day period. An elapsed time indicator is used to measure the total time that the sampler operated.

If a Moore and Gentry flow controller is being used a separate timer must be mounted:

- mount the 7 day mechanical timer on the inside of the door, directly beneath the flow controller and mount the elapsed time indicator so that it is easily viewed.

- Ensure that the timer does not interfere with door movement.

4.3 Flow Measurement

Air-flow is controlled by motor speed which is regulated with a mass flow controller. Airflow through the resin is maintained at 0.42 m^3 per minute at all times. Volumes collected are approximately 2500 m^3 over 4 days. Flow-rate readings, using a manometer and top-loading calibrator (orifice assembly) (Figure 4.4), are taken at the start and end of each sampling period. The average flow is used to calculate the sample air volume and is used to check flow controller operation.

The orifice assembly must be calibrated periodically against a primary standard as described in section 4.3.1. Under normal condition the flow controller will never need to be calibrated against a primary standard. However, the flow controller must be checked frequently and may require periodic adjustment as described in section 4.3.2.

4.3.1 Calibrating the Orifice Assembly

This section describes how to calibrate the orifice assembly and adjust flow rates to compensate for drift. The top-loading calibrator (orifice assembly) consisting of the calibration plate and the inlet restrictor with pressure tap, and manometer unit (Figure 4.4) are calibrated against a primary standard semi-annually, and a calibration curve plotted from the airflow versus differences in static pressure. The calibration curve thus produced is unique to each orifice assembly. The calibration points must lie within the normal operating range of the Hi Vol pump (0-0.84 m^3 per minute).

The calibration procedure is described below:

- Attach the orifice calibration assembly to the intake end of the positive displacement primary standard and attach a HiVol vacuum motor to the exhaust end of the primary standard. Use a voltage regulator to control power to the motor.

- Connect one end of a differential manometer to the differential pressure tap of the calibration unit and leave the other end open to the atmosphere.

- Attach one end of a second manometer to the inlet pressure tap of the primary standard and leave the other end open to the atmosphere. Measure the inlet pressure of the primary standard with this second manometer during each of the constant airflow measurements made below.

Placing the orifice before the inlet reduces the pressure at the inlet of the primary standard below 1 atm. It is necessary to measure the inlet pressure of the primary standard and make a correction for the increase in volume caused by this decreased inlet pressure.

- Operate the HiVol motor so that a series of 5-6 different but constant airflows (Q) (0-0.84 m^3 per minute) are obtained for definite time periods. Modify airflow by adjusting the voltage going to the motor. It is not necessary to record the voltage.

- Record the pressure readings of both manometers at each airflow. Record the barometric pressure and temperature at each air-flow.

- Calculate the corrected air volume measured by the positive displacement primary standard as follows:

$$V = (P_a - P_m)/P_a \times V_m$$

V corrected volume of air at the measured temperature and pressure, in cubic feet

P_a barometric pressure in millimetres of mercury

P_m difference in pressure between that measured at the orifice inlet and that measured at the primary standard inlet, in millimetres of mercury.

V_m volume measured using the primary standard, in cubic feet.

- Calculate the corrected airflow (Q_1) from the measured air volumes as described above. Plot each corrected value for airflow against the corresponding square root of the air pressure as recorded at orifice assembly pressure tap. This relationship should be linear. Outlying values result from error and must be repeated.

$$Q_1 = V/T$$

- Correct each flow rate (Q_1) to standard temperature (298°K) and pressure (760 mm Hg) as follows:

$$Q_{NTP} = Q_1((P_M/T_M) \times (T_{NTP}/P_{NTP}))^{1/2}$$

Q_{NTP} estimated flow rate at standard temperature and pressure

Q_1 corrected air flow

P_M barometric pressure at time of calibration

T_M absolute temperature at time of calibration

T_{NTP} standard temperature

P_{NTP} standard pressure

- Plot Q_{NTP} on the same graph as Q_1. This is the calibration curve and is to be kept on file in a three ring binder.

4.3.2 Adjusting Flow Rate

It is important that adjusting the flow rate in the field not be confused with calibrating the top-loading orifice assembly. It is not necessary to calibrate the flow controller or motor independently against a primary standard. The flow-rate is checked and recorded at the start and the end of every sampling period using the top-loading orifice assembly and a battery-operated digital manometer. Measured air flow should show less than 5% variation over the sampling period.

Airflow is adjusted only if manometer readings deviate by 10% from the expected reading as shown on the calibration curve for that particular orifice assembly. Never adjust airflow midway during a sampling period.

Setting or changing the flow-rate is done by simply adjusting the flow-selector potentiometer inside the flow controller. This is marked 'set screw'. Remove the cover of the flow controller to expose the flow control set screw. The correct screw is marked 'set flow'. Turn the set screw counter-clockwise to decrease flow and clockwise to increase flow. All flow controller adjustments must be recorded in the site log book and reported to the network coordinator.

The procedure for adjusting the flow rate in the field is described below:

- Obtain the following materials before checking flow rate and setting the flow controller to a specific setting:

  1 mass flow controller
  1 calibration orifice assembly with pressure tap
  1 manometer plus tubing

- Attach the pre-calibrated orifice assembly to the top of the filter horn (Figure 4.4). Ensure that the four butterfly nuts are tightened so that pressure is evenly placed on the filter holder. Turn the manometer on and connect the manometer tubing to the nipple.

- Turn the flow controller on to start the motor. Let the motor run for 5 minutes so that the flow can stabilize. Record the manometer reading in inches of water.

- If the manometer reading is incorrect, turn the flow control set screw

very slowly, making only small adjustments until the manometer gives a reading which corresponds to an airflow of 0.42 m^3 per minute as read from the calibration curve. Wait until the reading is stable (at least 15 min) before re-sealing and disconnecting the flow controller.

4.4 Changeover

This section describes how to collect exposed air cartridges and filters and install clean cartridges and filters in preparation for taking a sample. The entire sample changeover procedure should be performed while wearing cotton gloves or with bare hands. *Under no circumstances should changeover be performed while wearing plastic gloves.*

Hi Vol organics in air samples may be changed at any time the sampler is not operating i.e. inactive. It is not necessary to perform sample changeover on the same day each sampling period. It is usually most convenient to perform sample changeover between Monday and Friday of the week the sampler is scheduled to be inactive.

The sampler runs for 4 days (Monday to Friday) every second week. If the sample cannot be removed on the Friday (sampling end) it is necessary to stop the sampler from re-starting on the following Monday by removing the 'ON' switch during active operation of sampler. If the 'ON' switch is not removed the sampler will re-start at 8 am the following Monday. Consequently, each site must be visited at least weekly to alternately collect and deploy each sample and to remove the 'ON' switch.

When handling blanks are required the extra cartridge and filter thus designated are to be stored inside the Hi Vol housing for the duration of the sampling period and returned with the active samples.

4.4.1 Preparing for Changeover

To prepare for Hivol changeover requires essentially the same preparations and materials as needed to prepare for organics in precipitation changeover. Obtain

the following supplies to collect and replace a cartridge and filter in the field:

- Teflon squeeze bottle (methanol)
- Winchester methanol
- glass waste bottle with cap
- Kimwipes
- aluminum foil
- cotton gloves

4.4.2 Instrument Operation

This section describes how to check sampler operation and set the sampler up to collect an air sample:

- Insert the air flow sensor into the extension tube and secure it with a hose clamp. Check that it is securely sealed where it passes into the extension tube with a small silicone 'O' ring.

- Plug the motor into the flow controller outlet.

- Plug the flow controller into a power source. If the timer is not built into the flow controller the flow controller should be plugged into the mechanical timer. If there is a built in timer (some models) the flow controller is plugged directly into the main power source.

- *To start the sampler,* flip the ON/OFF switch in the mechanical timer to the ON position. The motor should start immediately. After a slight hesitation the motor speed will increase to a maximum speed before settling down at the pre-set rate. The motor speed may oscillate for about one minute before stabilizing at the pre-set rate.

- *To set the mechanical timer,* flip the ON/OFF switch in the mechanical timer to the OFF position. Loosen the screw fastening the 'ON' pointer to the circular dial showing the time and date. Slide the 'ON' pointer to 8 am Monday and tighten the screw. Similarly, slide the 'OFF' screw to the dial position corresponding to 8 am Friday. The

sampler is termed 'active' during the week in which sampling takes place.

4.4.3 Sample Collection

This section describes the procedures to collect exposed samples and measure the final flow rate. All activities are described in the order that they are performed.

4.4.3.1 Final flow rate

The final flow rate must be measured before removing the filter or cartridge. To determine the final flow rate for the exposed sample:

- Ensure that the instrument is turned off, lift the hood of the Hi Vol shelter and latch in the open position.
- Loosen the 4 butterfly nuts fastening the filter holder in place. Cover the filter holder with the calibration plate and thread the inlet restrictor onto the calibration plate.
- Turn the manometer on. Ensure that the manometer is set to read from 0 to 19.99 inches of water. Use the 'slow' setting. Let the manometer warm up for several minutes before zeroing.
- Push the tubing connected to the negative port of the manometer over the side nipple on the inlet restrictor. Check that the tubing is secure and that there are no leaks.
- Flip the ON/OFF switch on the mechanical timer to the ON position. Let the motor run at least 5 min. until the manometer reading stabilizes. Record the manometer reading in inches of water in the site log book and on the operator sheet (0.42 m^3 per minute corresponds to about 0.7" H_2O).

4.4.3.2 Active filter

To collect the active filter:

- Disconnect the manometer tubing from the inlet restrictor nipple and place the manometer to one side.
- Loosen the sliding ring between the funnel horn and outer cartridge holder.
- Lift the entire calibration unit plus the funnel horn out of the housing.

During windy conditions it may be impossible to remove the filter holder and change the filter without it tearing or blowing away. To prevent this carry the calibration unit and funnel horn to a sheltered area. If necessary a car will suffice.

- Place the entire unit on a horizontal surface with the outlet of the funnel horn down.
- Unscrew the funnel horn from the top of the cartridge holder. Place it on a clean piece of aluminum foil with the outlet of the funnel horn down.
- Loosen the 4 wing nuts fastening the filter holder in place. Remove the orifice assembly and filter holder to expose the filter.
- Place a clean piece of aluminum foil about 12" square on a flat surface sheltered from the wind.
- Carefully grasp one corner of the filter with Teflon-coated tweezers and lift. Place the filter on the piece of aluminum foil with the dirty side facing up. Extreme care is required to prevent filter tearing.
- Touching only the foil, fold the filter in half. Fold the aluminum edges until the folded filter is thoroughly sealed inside.
- Label the filter packet and place it between 2 sheets of cardboard. Identify the sample on the label i.e. '**Active Filter**'. Include the

sampling start and end dates and the **site name** on the label. It is not necessary to write the date of collection on the sample although this should be recorded on the field sheet.

4.4.3.3 Active resin cartridge

To collect the active cartridge:

- Lift the silicone gasket from the top of the 'active' cartridge holder and place it beside the funnel horn on a clean piece of aluminum foil.
- Remove the female transporting cap from the top of the fresh cartridge holder and securely thread the cap onto the inlet end of the 'active' cartridge holder while still inside the Hivol. Be careful not to turn the fresh cartridge holder upside down after removing the cap as the inner cartridge may fall out.
- Loosen the sliding ring between the bottom of the 'active' outer cartridge holder and the extension tube. Remove the 'active' cartridge holder from inside the HiVol. Place it upside down beside the funnel horn on the aluminum foil.
- Remove the silicone 'O' ring from the bottom of the 'active' outer cartridge holder. Place it beside the silicone spacing ring removed earlier from the top of the cartridge holder.
- Remove the male cap from the bottom of the fresh cartridge holder and thread the male transporting cap into the outlet end of the removed 'active' cartridge holder.
- Label the 'active' cartridge holder and store it with the active filter. Identify the sample on the label i.e. '**Active Cartridge**'. Include the **sampling start and end dates** and the **site name** on the label. It is not necessary to write the date of collection on the sample although this should be recorded on the field sheet.

4.4.4 Sample Installation

This section describes the procedure to install a new cartridge and filter, measure the initial flow rate and set the timer.

4.4.4.1 Fresh resin cartridge

To install a fresh cartridge:

- Remove the end-caps from a fresh cartridge holder.
- Slide a clean 'O' ring over the bottom rim of the fresh cartridge holder. The 'O' ring removed earlier from the exposed cartridge may be re-used after rinsing with acetone.
- Fit the fresh cartridge holder into the rim of the extension tube inside the sampler. Ensure that the 'O' ring is not displaced and that the gap between the cartridge holder and the extension tube is securely sealed. Tightly thread the outer cartridge locking ring down onto the extension tube to securely join the two pieces.
- Ensure that the surface of the inner cartridge is raised about 1/8" above the outer cartridge rim. If not, check that the two inner cartridge 'O' rings are not displaced.
- Place a gasket over the top of the cartridge holder so as to cover both the outer rim and the inner cartridge. The gasket removed earlier from the from the top of the outer cartridge holder may be re-used after rinsing with acetone.
- Thread the filter horn onto the inlet end of the cartridge holder so that pressure is placed on the inner cartridge as well as the rim of the cartridge holder and so that all connections are thoroughly sealed.

4.4.4.2 Fresh filter

To install a fresh filter:

- Remove the aluminum foil from the fresh filter and carefully centre the filter, rougher side up, on the supporting screen of the funnel horn (take care to only touch the filter with the aluminum foil).

- Cover the filter loosely with the filter holder before attaching the orifice assembly and taking the initial flow reading.

4.4.4.3 Initial Flow rate

- Determine the flow-rate using the orifice assembly and manometer as described in section 4.4.3.1. It may be necessary to re-set the mass flow controller by adjusting the potentiometer as described in section 4.3.2. 0.42 m^3 per minute +/- 5% is the acceptable range of operation.

After determining the initial flow:

- Remove the orifice assembly. Tighten the butterfly nuts located at each corner of the filter horn to fasten the filter holder down.

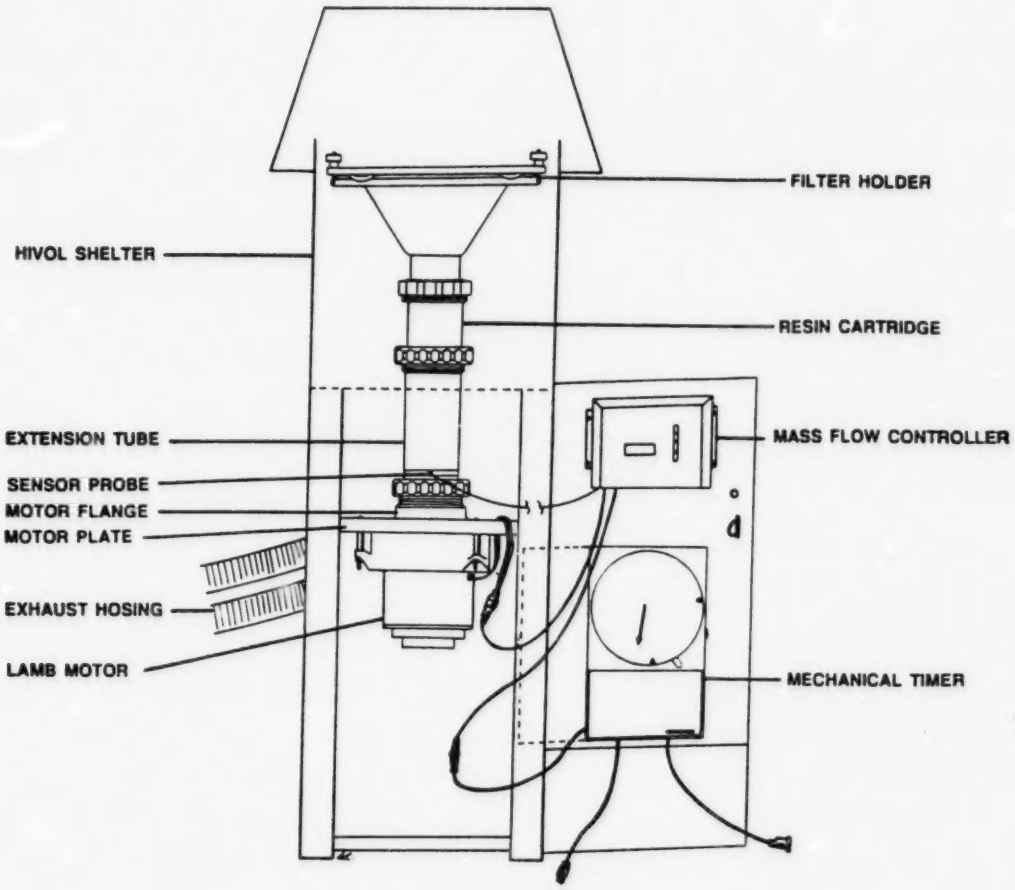
4.4.4.4 Timer

To set the timer:

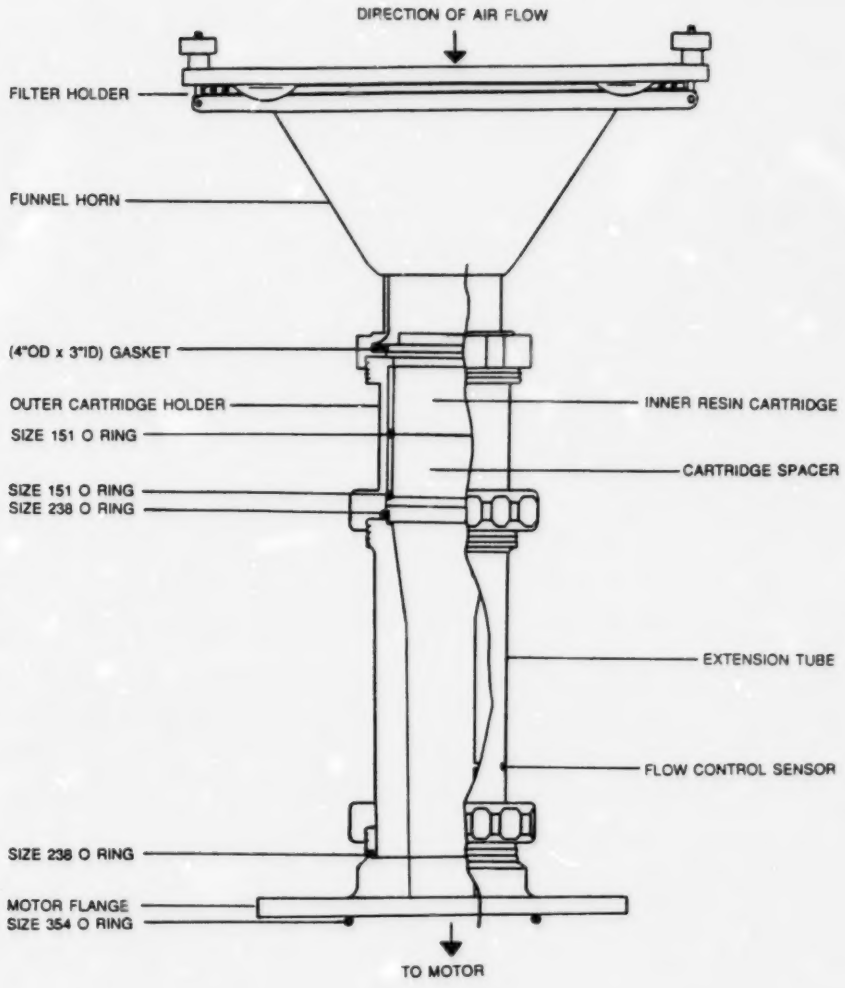
- Set the mechanical timer to start sampling at 8 am Monday and finish sampling at 8 am Friday by placing the on/off clamps at the appropriate settings on the timing dial.

- Ensure that the ON/OFF switch is set to the OFF position before leaving the sampler and that all connections are secure and all power cords are plugged into their proper receptacles,

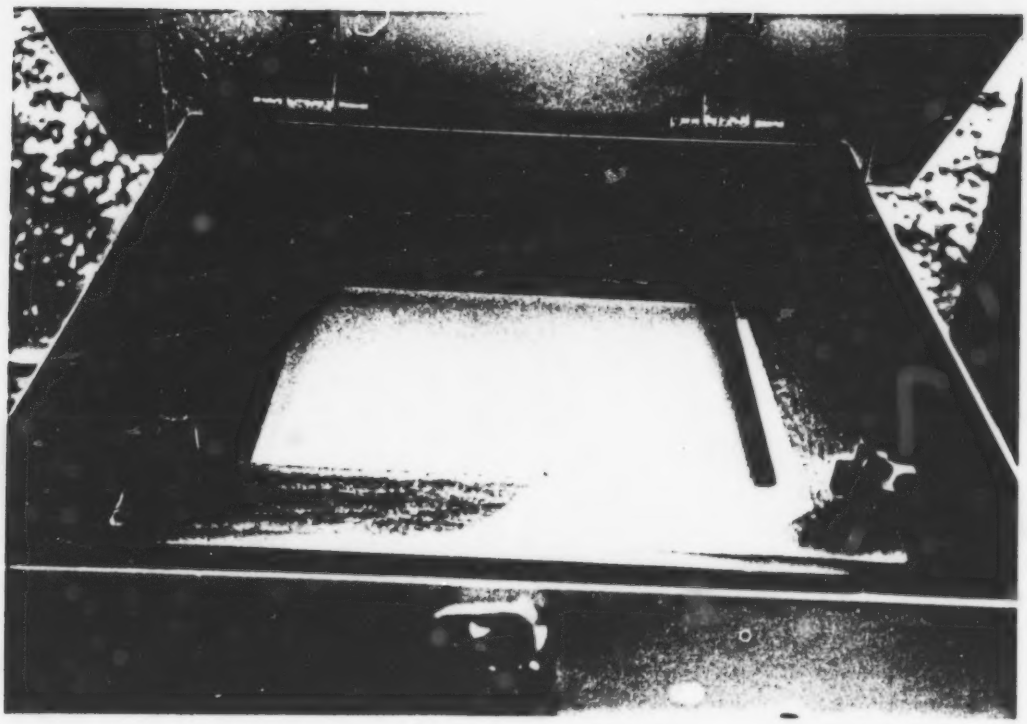
4.1: Diagram of a HiVol Air Sampler and Sampling Train.



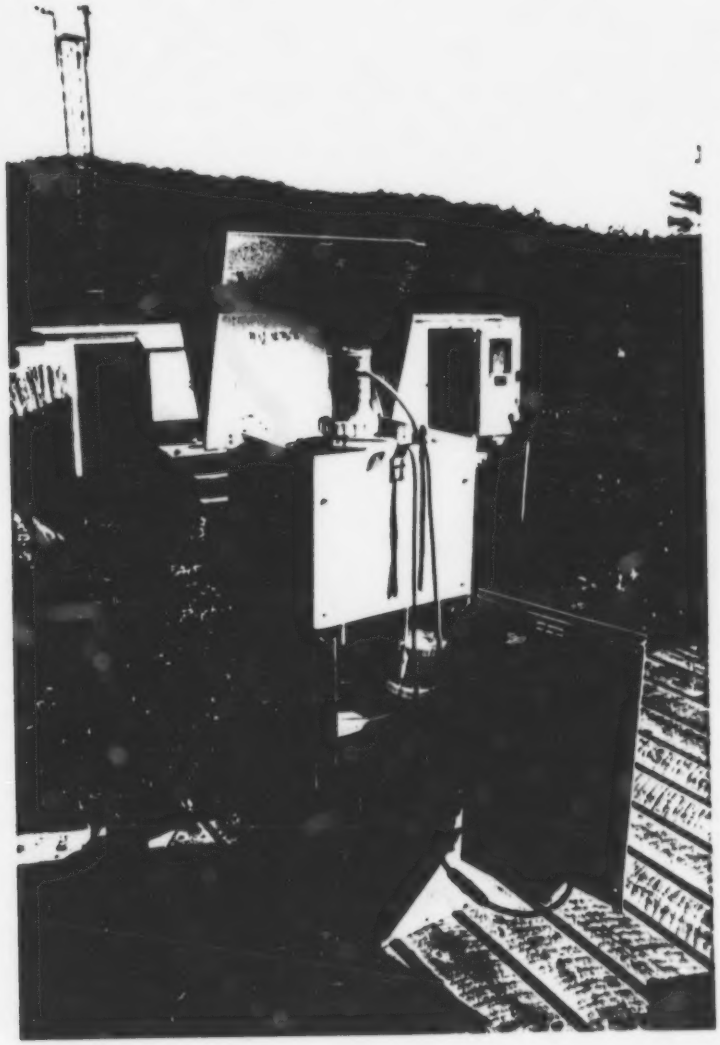
4.2: Internal Schematic of a HiVol Air Sampling Train.



4.3: Photograph of a HiVol Particulate Filter and Funnel Horn.



4.4: Photograph of a Calibration Assembly and Manometer in Position to Take a Flow Reading.



5 PREVENTIVE MAINTENANCE AND TROUBLESHOOTING

This chapter describes site and instrument checks, maintenance schedules and troubleshooting protocols for the organic deposition monitoring program. These schedules and protocols cover most instrument and sampling problems. Specific tools are required for maintenance and in order to make repairs and should be carried at all times.

- hammer
- vice grips
- crescent wrenches (various sizes)
- slot, Phillips and Robertson screwdrivers (various sizes)
- needle nose pliers
- adjustable pliers
- socket set
- wire stripper
- jewellers screwdrivers
- 2' carpenters level

It is important to note that problems may arise that cannot be resolved on-site. The site operator should be prepared, if necessary, to transport non-working samplers from the site to a place where repairs may be effected.

5.1 Site and Instrument Checks

This section describes the regular site inspection and individual instrument checks that should be performed on each site visit. The entire procedure should require less than one hour to complete per site. It may require more time if there are extensive problems or collocated samplers at the site. Troubleshooting problems is described in section 5.3.

General site and instrument inspections should be performed first to uncover obvious problems i.e.

- Check that all instruments are free of obstructions such as tall grass,

trees, shrubs and other monitoring instruments (2.5 x height rule).

- Check for loose power cords, vandalism etc.
- Clean the instrument housings by wiping the MIC B and HiVol housing exteriors and the underside of each MIC B hood with clean kimwipes and acetone.
- Record all instrument malfunctions and sampling problems and the date in the site log book and report such to the regional technician or network coordinator immediately.

Specific checks on individual instruments should be performed next as described in sections 5.1.1 and 5.1.2.

5.1.1 MIC B Checks

- Check hood operation.
- Check for precipitation pooling in the funnel.
- Remove any debris from funnel.
- Check for leaks in sampling train and cracks in welded joint in the base of the funnel.
- Check hood gasket fit on collection funnel.
- Check for over-full effluent bottle.

5.1.2 HiVol Checks

- Inspect and tighten each seal in the sampling train to ensure that there are no leaks.
- If the instrument has been in operation check the elapsed time

indicator to ensure that elapsed sampling time is correct and there have been no interruptions.

- Check that there is power to the HiVol motor and that the HiVol will operate properly by briefly turning the instrument on (Use the ON/OFF switch in the mechanical timer).

- Check and record the flow rate while the instrument is turned on with the manometer and calibration plate as described above.

5.2 Scheduled Servicing

This section describes all scheduled instrument servicing. All scheduled maintenance and servicing should be performed after checking instrument operation.

5.2.1 MIC B Scheduled Servicing

MIC B scheduled maintenance is as follows and should be performed semi-annually.

- Replace hood gasket (may need replacing every 3 months).

- Inspect and replace limit switches.

- Lubricate hood drive motor.

- Check the clutch adjustment (15 to 20 lbs) using a spring gauge.

- Check the small and large sprockets for correct alignment. Ensure that the set screw on the small sprocket is tight. Loose set screws may cause the hood to drop.

- Check the transformer output (12.5 to 15.0 Volts AC).

5.2.2 HiVol Scheduled Servicing

The only regular, scheduled HiVol maintenance is as follows:

- Remove the motor shroud and change the brushes every three months. Run the motor at 1/2 speed for 30 minutes to seat the new brushes.
- Replace armatures annually
- Perform a multipoint calibration of the top loading calibrator and flow controller semi-annually. This is done at Chesswood Drive.

5.3 MIC B Troubleshooting

Troubleshooting is the process of recognizing instrument malfunctions as they occur and rectifying them. Section 5.1 described the process of problem identification. This section describes how to deal with operating problems or instrument malfunctions uncovered.

5.3.1 Mechanical Problems

Instrument malfunctions will usually affect hood movement and gasket fit. Step-by-step troubleshooting protocols to eliminate instrument malfunctions are described below.

The following supplies should be on hand to effect any repairs as occur:

- 220 K ohm resistor
- sensor grids
- 2 amp. slo-blo fuses
- limit switches
- P.C. boards

5.3.1.1 Hood Movement

The sampler hood should open after touching both sides of the sensor with a moist finger or during a precipitation event, and close within 1 min 30 sec (+/- 30 sec) of touching with a wet finger or a precipitation event ends. If the hood does not operate within these specifications proceed with the following series of checks until the problem is identified and solved:

- Ensure that the instrument is plugged into a ground receptacle and turned on.

- Test the ground fault located on the ground receptacle. Reset the ground fault if tripped.

- Check the main electrical panel for tripped switches, reset if necessary.

Turn the instrument off and remove the splash cover before proceeding:

- Unscrew the fuse from inside the motor housing. Check to see if the fuse has blown. A blown fuse will appear blackened and burnt. Replace the fuse if blown.

- Check the sensor grids located on the sensor arm. The sensor grids should be warm to the touch. Replace if burned out. Ensure that the sensor grid wires and connections are thoroughly insulated and secure.

- Check the limit switch inside the instrument. The limit switch should make firm contact with the limit screw when the hood is completely open. Adjust or replace the limit switch if necessary.

- Check the P.C. board located inside the instrument, replace if necessary.

If everything appears normal and all components have been checked or replaced, but the moisture sensors are not registering a response to rain/snow events it may be necessary to adjust the instrument sensitivity to moisture as follows:

- close the contact surface on the sensor grid with the 220 K ohm

resistor. The sampler should activate exposing the collection vessel. If the hood still does not activate, turn the sensitivity trim pot on the printed circuit board clockwise. If there is no response, turn the trim pot counter-clockwise until the hood activates. Leave the trim pot in this position.

5.3.1.2 Hood Gasket

If the hood gasket does not form a tight seal over the collection vessel or hood movement is not smooth:

- check that the hood gasket is centred properly.
- Turn off the instrument and remove the splash cover.
- Check the chain tension and adjust if necessary. There should be about 1/4 to 1/2" of play in the chain. Overtightening causes 'pings' while undertightening causes the hood to drop.
- Ensure that the sprocket set screws are tight since this may cause the hood to drop.
- Check the limit screw adjustment to ensure that firm contact is made with the limit switch when the hood is fully open. Poor contact may cause the hood to swing back and forth and will cause the arms of the hood to break.

5.3.2 Sampling Train Problems

Common sampling train problems include precipitation bubbles in the sampling train, clogged or dry filters, too tight resin cartridge fittings, closed stopcocks or clogged spaghetti tubing. All of these problems affect flow and usually reveal themselves in precipitation pooling in the funnel or partial draining of the collection column.

If precipitation is pooling in the collection funnel:

- Adjust the end of the spaghetti tubing in the mouth of the effluent bottle above the filter pack. This keeps the filter moist and inhibits excess draining of the cartridge.

- Turn the flow control knob of the drip adaptor at the base of the cartridge clockwise to increase flow.

- Dislodge any precipitation bubbles trapped in the neck of the funnel above the filter pack with a blunted, methanol rinsed cable tie. Take extreme care not to puncture the filter when poking the cable tie into the funnel opening.

- Loosen the resin cartridge from the column adaptor slightly (about 1/2 turn).

If performing the above actions does not increase the flow, the particulate filter must be changed. This must be done carefully so as not to spill any sample.

- Place a clean overflow bottle underneath the collection funnel.

- Close the flow-control spigot.

- Unscrew the filter pack from the Teflon adaptor and quickly position the spare effluent jug to catch the precipitation. Collect all of the pooled sample.

- Disassemble the filter pack, remove the filter and wrap it in aluminum foil.

- Put a new filter in the filter pack, reassemble the filter pack and reconnect it to the column adaptor.

- Pour the collected precipitation back into the funnel.

If the XAD 2 resin is not submerged and is partially or completely drained (i.e. through improper positioning of tubing, blockage in the sampling train or if a leak has developed),

- the resin cartridge must be topped up with distilled/deionized water. If water has pooled in the funnel it is simply necessary to identify the cause of the pooling and correct it as described above. The pooled precipitation will flow into the cartridge submerging the resin.

If the funnel is empty and the cartridge is partially or completely drained:

- add a small amount of distilled/deionized water to the funnel and let drain through sampling train.
- loosen the collection column slightly.
- Ensure that the Teflon tubing is properly looped above the filter, as described above.
- If the water still does not drain into the column, gently loosen the column further.
- If the excess added water does not drain from the cartridge there is probably a blockage in the sampling train. Repeat the procedures above (for draining pooled precipitation) to remove the blockage
- Continue adding water until the column is full and there is enough water in the funnel adaptor and reservoir to keep the particulate filter moist. Sufficient water has been added when drops start to fall from the Teflon tubing into the effluent bottle.

5.3.3 Temperature Control

The internal sampler temperature should be maintained between 5 and 10 °C during the winter in order to minimize evaporative losses of the sample and prevent freezing of the sample.

If there is no heat inside the sampler housing, or the cartridge or jug is frozen, check to make sure that heaters (if present) are working as follows:

- ensure that the heater extension cord is plugged into the ground receptacle.

- Check the ground fault and reset if necessary.
- Turn the heater thermostat control up. This should produce an immediate response.

Damaged and broken resin cartridges are to be removed, reported to the network coordinator and immediately returned. Do not discard damaged cartridges.

If the internal temperature of the sampler is too high, adjust the thermostat down.

5.3.4 Effluent Bottle

If the effluent bottles are full they must be replaced immediately.

For cartridge sampling

- gently pull the Teflon tubing from the mouth of the effluent jug;
- lower the jack and remove the effluent bottle from the jack without disturbing the sampling train or spilling;
- replace with a clean, empty effluent jug as described above;
- seal and label the effluent jug as such and include the date of removal and the site name;

For jug sampling

- carefully remove the aluminum foil from around the mouth of the bottle and the funnel drain;
- lower the jack and remove the spiked effluent bottle from the jack without spilling;
- replace with a clean, empty effluent jug as described above;
- seal and label the spiked effluent jug as such and include the date of

removal and the site name;

5.4 HiVol Troubleshooting

Problems with the high volume air sampling equipment should occur infrequently. The most likely sources of sampling interference are likely to include:

- power failures and ground fault interruptions,
- sampling leaks due to poor seating of the cartridge and cartridge holder during sample changeover or to motor vibration over time,
- drift in the flow rate cause by incorrectly locating the flow controller sensor or a mechanical problem with the flow controller,
- motor failure due to excess wear on motor brushes.

All of these problems will reveal themselves by affecting the flow rate or time of sampling

.

5.4.1 Air Flow

If the rate of flow is not as pre-set, identify and eliminate the problem in the following order of priority. Eliminate leaks, adjust the flow controller, change motor or replace the motor brushes. Note that high winds and rapid changes in wind speed and direction may interfere with the measurement of flow and may cause the motor speed to vary.

Eliminate leaks as follows:

- remove the filter horn, and firmly tap the cartridge down into the cartridge holder. Replace the filter horn and secure.
- Loosen the hose clamp holding the sensor probe in place and slide the probe out of the extension tube, inspect the probe for damage and carefully replace the sensor making sure that the opening in the side

of the probe near the tip is facing directly upwards (ensure that there is a small 'O' ring at the base of the sensor probe used to seal the opening into which the probe fits),

- Check that all 'O' rings are seated securely and connections are tight.

If there is no improvement in flow:

- Check that all electrical connections are correct and secure.
- Adjust the flow controller by turning the set screw. *Do not adjust the flow-rate in the middle of a sampling period.* Simply note the discrepancy and record the flow-rate.

If the flow is oscillating:

- disconnect the motor from the flow controller and reconnect the motor directly to the main power supply or to a power transformer (VARIAC).
- if flow continues to oscillate check all seals and electrical connections to ensure that they are secure.
- change the motor brushes if the flow continues to oscillate.

If it is not possible to stabilize flow the flow controller must be replaced and returned for repair.

5.4.2 Timer

If sampling times are incorrect:

- check that the 'ON/OFF' switch was removed on schedule.
- check the ground faults for interruption.
- check timer to ensure operation.

6 SAMPLE HANDLING AND SUBMISSION

After collecting the samples in the field, the samples must be packed in coolers and prepared for transport to the laboratory. This section describes the procedures to label, ship and submit exposed samples.

6.1 Labelling and Documenting

Before shipping the samples to Toronto the samples are labelled and recorded in the site log book by the site operator. An operator sheet is completed by the site operator for all samples collected. RFA/Field sheets are filled out and duplicate field sample numbers are affixed to the sample and the RFA/Field sheets by the regional technician. Each site operator must ensure that:

- all of the samples recorded on each RFA/Field Information sheet have a common **station ID** and **sample collection date**,
- **field sample numbers** and **sample descriptions** on the RFA/Field Information sheets correspond to those on the samples.

The procedure to label and document samples is as follows:

- Attach a sample label to each sample (including the handling blank) while on-site.
- Complete the following information on each label with a pencil. Do not use a ball-point pen to write on the label. Do not write on the label or directly on any sample with an indelible marker because this may contaminate the sample through volatilization of organic compounds from the ink.
    - Station Name and Number, e.g. Toronto Island, #3201
    - Name of the Person who Collected the Sample,
    - Sample Type, e.g. Active Resin Cartridge
    - Date of Sample Collection.
- Complete each sample collection entry on the operator sheet while on-

site. Use one operator sheet for all samples collected on the changeover. Keep all operator sheets permanently in a three ring binder. Alternatively a hard-cover field notebook (site log book) may be used to record all pertinent information while on-site for later transcription to an operator sheet or directly to an RFA/Field sheet.

- Attach a field sample number to each sample and affix the duplicate field sample numbers to the RFA side of the RFA/Field sheet. The label and field number must be attached directly to the cartridge not to the cartridge travelling tube.

- Transfer all sample collection information from the field note book or operator sheet to an RFA/Field sheet. The acetone final rinse will have the same field sample number as the cartridge. Copy each field sample number onto the field submission side of the sheet. Complete one RFA/Field sheet for all of the samples collected under the same program code (see chapter on network documentation).

- Ensure that when completing the RFA/field submission sheets, matching numbered stickers are attached to both the RFA/field submission sheet and the sample.

6.2 Shipping Procedures

The procedures for shipping fresh samples to a site before changeover or for returning active cartridges or filters after changeover are essentially the same for air and precipitation sampling. Samples are not to be shipped on a Friday to avoid samples sitting in a warehouse over the weekend. The objectives of this section are to ensure that standard shipping procedures are followed so that:

- all filters, cartridges and jugs arrive at their destination uncontaminated and intact,

- time in transit is minimal

After Changeover the site operator will:

- Safely pack the active precipitation cartridge, filter (not the filter

pack), used spaghetti tubing and final rinse bottle in coolers with sufficient packing material to ensure that no samples are broken. Enclose the completed 'RFA/Field submission sheets' in a plastic bag and place in the shipping container with the samples. Ship the active samples in the same coolers in which the fresh samples arrived. Do not ship samples in other container types.

- Seal the active air cartridge holder securely with both travelling caps. Pack each field blank air cartridge holder the same way. Ensure that each air filter is folded with the surface in and securely sealed in aluminum foil and that a label is completed and attached to each filter sample. Enclose the completed 'RFA/Field submission sheets' in a plastic bag and place in the shipping container with the samples.

- Ensure that all solvents being shipped are accompanied by all necessary shipping or WHMIS documents. This includes all acetone and dichloromethane final rinse samples. Methanol must be similarly documented if being shipped.

- Arrange for a courier to ship the samples to the laboratory if it is impossible to transport them directly. The shipping address is:

  Ontario Ministry of the Environment,
  Central Stores,
  125 Resources Rd.,
  Toronto, Ont.,
  M9W 5L1

  attention: APIOS shipping technician

6.3 Submission

The samples and RFA/Field sheets are delivered to the laboratory sample reception area. There they are processed every Monday and Thursday for submission by the APIOS processing technician who will:

- Check each sample against the appropriate RFA/Field submission sheets by matching field sample numbers on the sheets with those on

the samples.

- Note missing samples and trace them immediately.

- Correct field sample numbers on the RFA/Field sheets that do not match the numbers on the samples. Make all corrections on the RFA/Field sheets. Do not attempt to change the numbers on the samples.

- Scan RFA/Field submission sheets for possible errors or omissions and sort the RFA/Field sheets in order of date. Contact the regional technician to verify that any changes or corrections are valid.

Errors and omissions to note include:

  - missing data e.g. sample volume, instrument serial number

  - incorrect data, e.g. wrong stationid, replicate sampler number, or instrument type, incorrect program codes, or number of containers sent,

  - inconsistent entries for samples collected from the same instrument,

- Group the samples and RFA/Field sheets on the basis of program codes into separate submissions. Air samples are grouped into one program code and precipitation samples into another.

- Prepare a single covering submission form for all of the samples to be submitted under a program code (e.g 020020402: Organics-in-Air samples).

- Place a sequentially numbered bar code submission number sticker on the covering submission form. This allocates the submission number for that set of samples. All of the RFA/Field sheets and samples grouped under a program code are submitted under the same submission number and with the same submission covering sheet.

- Copy the submission number from the covering submission bar code

sticker to the front and back of each RFA/Field sheet in the submission.

- Record all of the samples in each submission in a submission record book.

- Make 2 photocopies of the covering submission form and the "Request for Analysis" side of all of the RFA/Field sheets in the submission.

- Make 1 photocopy of the Field information side of each RFA/Field sheet to be submitted.

- Place the correct stationid sticker in the assigned box on the top of each photocopied RFA sheet and Field information sheet in the submissions.

- Place corresponding stationid stickers on the both sides of the original RFA/Field sheets. Single submissions may contain samples from different stations and care should be taken to ensure correct labelling of the RFA/Field sheets.

- Take the samples plus documents to the appropriate laboratory for analysis where a technician will record all submissions in a log book in each lab, assign lab sample numbers, and enter the samples into the laboratory information system (L.I.S.).

- Submit 1 complete set of photocopied RFA sheets with their covering submission sheet to sample reception for data entry into LIS.

- Submit 1 set of Photocopied RFA sheet and the set of photocopied Field sheets to the organics lab with the samples.

- Return the original RFA/Field sheets to the head office data clerk for filing.

7 NETWORK DOCUMENTATION

Throughout this manual network documentation and record keeping procedures have been reported where appropriate. The objective of this chapter is to review network documentation and the record keeping process and simplify where possible.

7.1 Site Log Book

A hard-cover log book is to be continuously maintained on-site by the operator to provide a written record of samples collected, measurements, instrument checks and problems, and any other pertinent observations. Records of all site visits should be recorded in the site log book, not simply site changeovers.

The following information must be recorded in the log book on each site visit. If there is an on-site operator this information should be entered daily.

- Date
- Time of visit
- Status of instrument operation e.g. summary of problems, required maintenance, blown fuse etc.
- Sample field data and comments e.g. sample removed and replaced, filter clogged and replaced or cartridge broken, evidence of power interruption, poor flow control etc.

In addition to the above information the site operator must record the following information in the log book on changeover:

- Sample changeover (start/finish times) if applicable
- HiVol manometer reading using the calibration orifice if recorded.
- Effluent volume if applicable
- Total lovol flow volume if applicable
- Lovol flow rate if applicable
- Nipher solution depth
- Nipher solution temperature

Any changes to the site or instrumentation should also be recorded. This includes:

- Nipher gauge charging
- Addition or removal of instruments.

Observations which might affect the sample are very important and should be recorded at all times. Examples include spraying of pesticides or construction activity in the vicinity of the sampler. Thorough screening of the data often requires reference to the field observations in order to explain a particular analysis result.

7.2 Operator Sample Record Sheet

The operator sample record sheets contain all of the relevant sampling information and are to be completed by the individual collecting the samples upon sample collection. A copy of an operator sheet is shown in Figure 7.1. The sheets are self explanatory. Recording all essential information immediately will minimize any chance of error. All operator sample records remain in the possession of the site operators unless requested by the network coordinator. They are to be kept on file in a three-ring binder.

Multiple samples may be recorded on a single operator sample record sheet if collected at the same time from the same site and ancillary data are common to all samples entered on the operator sheet. 'Operator Sample Record Sheets' also need to be filled out for field blank and active samples or samples collected from a collocated sampler.

7.3 Request For Analysis/Field Information Sheets

Each sample collected must be allocated a field submission number and recorded on an 'RFA/Field Sheet' as described in chapter 6. Completed examples of precipitation and air 'RFA/Field' sheets are shown in Figures 7.2 to 7.5. This section describes how to complete the RFA/Field sheets. Extreme caution must be taken when completing the RFA/Field sheets to ensure that the sample numbers correspond to the field sheet numbers.

The RFA/Field information sheet combines the previous LIS RFA form and toxic

organics field sheet into one double-sided form. Each form may accept up to 8 samples. The format of the front of the form (RFA side) is common to all samples. The format of the back of the form is specific to air samples (SISTYPE =081) or precipitation samples (SISTYPE = 061).

When completing RFA/Field sheets, multiple containers are allocated the same sample number. The sample number must be handwritten on one of the samples because there are only two copies of each field sample number sticker.

The RFA/Field Information sheets are meant to be used on a 'one-per-instrument' basis. Do not:

- use more than one RFA/Field Information sheet to document multiple samples collected from a single instrument during a changeover, or
- list samples from different instruments on the same RFA/Field Information sheet.

Each site operator will be supplied with RFA/Field sheets prior to each changeover. The operators will keep photocopies of the front and back of the forms on file and send the original form with the sample(s) to the submission technician.

Complete both sides of the RFA/Field sheets according to the following criteria:

Note: * Fields marked with a single asterisk to be filled in by field technician (site operator).

** Fields marked with double asterisks to be filled in by APIOS submitting technician.

7.3.1 Side 1: Request for Analysis

Submission Number** The submission number is assigned using bar codes in sequential order with no duplicates by the APIOS

submitting technician. The submission number on the RFA sheet is copied from the covering submission form. AT submission numbers will be supplied **only** by the sample submission technician at LSB. The sample submission technician will check and initial each RFA/Field Information sheet before submitting the samples for analysis.

Stationid

The stationid is a valid active APIOS 14 digit station code used to indicate instrument type and the replicate number of the instrument at a particular APIOS station. The code is pre-printed on the RFA sheet.

e.g. 00000025003012 indicates the collocated hivol at Dorset. Instrument Type = '25' = Hivol. Station = '301' = Dorset. Replicate Number = '2' = 2nd

Program Code

The program code is a valid 9-digit LIS/SIS program code preprinted on the form. Currently defined program codes used in the Toxic Organics network are:

020020401	Toxic Organics in Precipitation
020020402	Toxic Organics in Air
020020501	Toxic Organics in Precipitation/Special Study
020020502	Toxic Organics in Air/Special Study

Regular network samples are submitted under the first two program codes only. Studies requiring the use of the Special Study program codes must be arranged through prior consultation with the database coordinator.

Instrument Serial No. *

The recorded instrument serial number is the MOE inventory number. If the instrument is replaced due to equipment failure, the old stationid will be kept, and the new serial number will be recorded on the RFA to indicate that the instrument has been replaced. HiVols are arbitrarily allotted numbers to be entered in this column.

Station Name — The station name is the APIOS station location as indicated in the 14 digit stationid and is preprinted to correspond with the stationid.

Instrument Type — The instrument type e.g. MIC Type B. This section will be pre-printed to correspond with the stationid.

Project/Subproject Description — A description of the project and subproject for which the data is being collected, to be preprinted on the form.

Remarks ** — Record lost samples, or samples not submitted.

Technician * — Initials of field technician.

Operator * — Initials of field operator, if applicable

Field Sample Number * — The Field Sample Number column will contain the 5 or 6 digit sample number sticker. The sticker number must match the field sample number written on the field data section on the back of the form and the sticker number on the corresponding sample containers. The sample containers(s) are described under the column labelled "DESCRIPTION" and must match the codes assigned on the back of the form with respect to collector type, sample matrix, QA/QC code, and filter/precipitation type.

LIS Sample Type — The LIS sample type must be a valid 2 letter code, defined and assigned by the LIS. These codes will be pre-printed on the form.

Lab Sample No. 1/ Lab Sample No. 2 — The lab sample numbers are for laboratory internal use only and must remain blank.

Containers Sent/Missing The containers sent is the number of containers expected for a particular sample. This information will be pre-printed on the form. The containers missing area is for internal laboratory use only, and must remain blank.

Sample Removal Date/Time* The sample removal date and time are copied from removal date/time written on the back of the form.

Lab Remark 1/ Lab Remark 2 The lab remark codes are for laboratory use only and must remain blank.

Stationid The stationid column will contain the stationid code from the top of the page. This code will be preprinted.

Description The description of the sample will be pre-printed to correspond with field data codes on the back of the form. For special study samples, or for changes in the regular network sampling protocol, the network coordinator will be required to advise the database coordinator of the required changes so that the forms may be changed accordingly.

Test Group ** The test group column will contain a valid test group name pre-assigned by the lab. The test group indicates the target compounds for which an analysis is being requested. Additions and deletions of test names to or from the test group will be indicated under the adjacent column marked '+/-', with additions and deletions clearly indicated by a plus or minus sign. Additions and deletions are made only by the APIOS submitting technician.

7.3.2 Side 2: Field Data

Submission Number ** The submission number is carried over from the front of the form.

Program Code The program code is pre-printed to match the program code on the front of the form.

Instrument Type The instrument type is described as on the front of the form. This information will be pre-printed.

Stationid The stationid is pre-printed to match the front of the RFA.

SIStype The SIStype indicates whether the sample is an air sample (081) or a precipitation sample (061). This information will always be pre-printed on the form.

Station Name The station name will match the station name pre-printed on the front of the form. This area will also be pre-printed.

Field Sample Number * The sample number must match the sticker on the corresponding container(s). Care must be taken to ensure that the sticker numbers are transcribed to the correct line describing that sample, as indicated by the collector type, sample matrix, QA/QC code, and filter/precipitation type on the back of the Request for Analysis form, and by the Description on the front of the Request for Analysis form.

Sample Removal Date/Time * The date (day/month/year) and time (hr/min) at which the sample exposure was **terminated**. This information must then be transcribed in full to the front of the form.

Initial Date/Time * The date/ (day/month/year) and time (hr/min) at which

the sample exposure was initiated.

Standard Period — The expected sampling duration of the sample according to a predetermined schedule established by the network coordinator. This code will be pre-printed on the form.

Collector Type — The physical container in which the sample is collected and submitted to the lab. For regular network samples, this code will be pre-printed on the form received by the field technician.

Sample Matrix — The material used to collect the sample, for example, a filter medium or a rinse solvent. This code will be pre-printed on the form received by the field technician.

QA/QC Code — The QA/QC code is used to distinguish regular active samples from various quality assurance/quality control samples. This code will be pre-printed on the form.

Field Comments * — In capital letters, indicate codes that describe the field conditions under which the sample was collected. It is important to note factors affecting the flow volume for air samples, or the collected sample volume or gauge depth for precipitation samples. It is also important to note signs of apparent or evident contamination. Note that there are two different series of codes for air and precipitation samples, respectively.

Field Remarks * — Handwritten sample remarks by the field technicians must be included on the field information sheets. These help the sample submission technicians to correct poorly labelled or incorrectly coded samples.

Field remarks should note:

- Additional or unusual samples collected as part of a special study must be noted on all field information sheets. All procedural changes must

also be specified.

- Any samples that are collected but not submitted should also be noted on all field information sheets. All such samples should be allocated an individual field sample number and be recorded on a separate field information sheet. The reason for not submitting the sample must be noted (e.g. cartridge frozen and broken, not submitted)
- The number and type of sample containers submitted with the sample, if applicable.
- All instrument malfunctions or nearby activities that might affect sample quality. If there has been an instrument failure that could affect the measured flow volume, or collected precipitation volume, briefly note this in the remarks section.
- Effluent volume.
- XAD-2 resin batch number and date of preparation (if available).
- Solvent name and volume used for final rinse.
- If there is evidence of sample contamination, describe the nature of the contamination. Describe any field conditions that may have affected the sample.

7.3.2.1 Fields for Air Samples

Filter Type The filter type code will be pre-printed on the form.

Corrected Flow Volume * The flow volume, calculated from the initial and final manometer readings, and corrected by the appropriate calibration factor, in litres. This will be a 7-digit number.

Initial/Final Manometer Readings * Enter the initial and final manometer readings

Elapsed Time * Indicate the elapsed time, in hours and fractions of

hours, from the instrument timer.

Batch Numbers *

Indicate, where applicable, batch numbers for the sample matrix.

Flow Controller Number *

Indicate the flow controller serial number for the flow controller currently installed on the instrument.

Calibration Orifice Plate Number *

Indicate the number of the currently installed calibration orifice plate.

7.3.2.2 Fields for Precipitation Samples

Precipitation at Exposure/ Removal *

Indicate with a '1' in the appropriate column if it was precipitating at the time of sample exposure or removal.

Precipitation Type

The precipitation type will be pre-printed on the form.

Gauge Depth *

Enter the gauge depth in millimetres, corrected for temperature.

Gauge Type *

Enter the appropriate gauge type code from the code list. If there is no installed gauge at the site, enter a gauge type code of '5'.

Precipitation Start/ End Times *

Daily samples only.

Total Sample Volume *

Enter the total volume, in millilitres, of precipitation collected between the sample exposure and removal date/times. This is equivalent to the 'effluent' volume.

The Total Sample Volume is exclusive of any additional volumes of rinses.

Rinse Volume *

If the collected sample is a rinse sample, enter the amount of solvent, in millilitres, used to rinse the instrument. This volume should not exceed 500 ml.

Cumulative Measured Gauge Depth/Supplemental Gauge Depth *

Enter the measured cumulative gauge depth, corrected for temperature. If the regular gauge depth is unobtainable, but a collocated gauge measurement is available, enter the replacement depth, in millilitres, under the column 'supplemental' and indicate the source of the measurement, (e.g. AES gauge, belfort gauge, etc.), under the column labeled 'HOW?'.

Degrees Centigrade *

Enter the temperature in degrees centigrade.

7.4 Laboratory Submission Covering Form

When the original RFA/Field sheet is received at LSB, a covering submission form is prepared for the RFA sheet. Submission number bar codes are attached to the front of each covering submission form. The submission number is transcribed to the front and back of each RFA/Field sheet in the submission.

The processing technician at Resources road will copy the submission number to the covering submission form and to the front and back of the RFA/field sheet and submit the samples to the lab.

Regional location(s), type of sample, organics deposition program code, date submitted to lab, and a listing of field sample numbers are all recorded under the submission number in the record book. Air and precipitation samples are to be given separate laboratory submission numbers and submitted separately. The corresponding field submission sheets are also assigned this laboratory submission number.

7.1: Operator Sample Record Sheet

OPERATOR SAMPLE RECORD SHEET - TOXICS PROGRAM

Station _____ Operator _____ Technician _____

MIC 'B' Organics in Precipitation

Sample start (date,time) _____
raining or snowing? _____

Sample end (date,time) _____
raining or snowing? _____
Effluent volume (mls.) _____

Final rinse volume (mls.) _____

Blank taken? _____

Time sampler switched on _____

Comments: (sample spilled, filter clogged, etc.)

Instrumentation Problems: (fuse blown, arm broken, GFI tripped, etc.)

When did malfunctions occur?
from _____ To _____
from _____ To _____

Missed events? _____

MIC 'B' Dioxins in Precipitation

Sample start (date,time) _____
raining or snowing? _____

Sample end (date,time) _____
raining or snowing? _____

Final rinse volume (mls.) _____

Blank taken? _____

Time sampler switched on _____

Comments: (sample spilled, jug overflowed, etc.)

Instrumentation Problems: (fuse blown, arm broken, GFI tripped, etc.)

When did malfunctions occur?
from _____ To _____
from _____ To _____

Missed events? _____

Hivol Organics in Air

Sample start (date,time) _____

Sample installed (date,time) _____
raining or snowing? _____
Manometer reading ($^{\prime}H_2O$) _____
Counter reading _____
Air temperature (°C) _____
Air pressure (torr) _____

Blank taken? _____

Sample end (date,time) _____

Sample removed (date,time) _____
raining or snowing? _____
Manometer reading ($^{\prime}H_2O$) _____
Counter reading _____
Air temperature (°C) _____
Air pressure (torr) _____

Comments: (filter torn, insects on sample, etc.)

Instrumentation Problems: (GFI tripped, timer malfunction, motor burned out, etc.)

When did malfunctions occur? From _____ To _____

Events missed?

Nipher Gauge Measurements: Depth (mm.)_____ Gauge Recharged?_____
Temperature (°C)_____ Recharge Depth (mm.)_____

Supplies Needed: (kimwipes, filters, etc.)

7.2: Precipitation Request for Analysis and Field Information Sheet (Side 1)

APICS REQUEST FOR ANALYSIS WEEK OF: 05 09 89

SUBMISSION #
ATC_____

STATION ID
00000 00800 3011

PROGRAM: 02002 0401

INSTRUMENT SERIAL NO.: MIC B 7702

STATION NAME: DORSET INSTR TYPE: MIC TYPE #1

PROJ/SUBPROJ DESCRIPTION: TOXIC ORGANICS IN PRECIPITATION

REMARKS: _____ TECH: J.P. VARTO OPER: J.P. VARTO

	FIELD SAMPLE NUMBER	SMP TYP	LAB SAMPLE NO. 1	LAB SAMPLE NO. 2	CONT S/M		DATE DD-MM-YY	TIME HH-MM
1	106476	Px			1		13-06-89	1210
2	106477	Cx			2		13-06-89	1200
3	106478	Fx			1		13-06-89	1200
4	106479	Cx			1		13-06-89	1200
5							" "	"
6							" "	"
7							" "	"
8							" "	"

	REM1	REM2	STATIONID	DESCRIPTION	TEST GROUP	+/-
1			00000 00800 3011	FILTER ACTIVE	G-PAAOC	
2			"	CARTRIDGE ACTIVE	"	
3			"		"	
4			"		"	
5			"		"	
6			"		"	
7			"		"	
8			"		"	

Use one Station ID per form.

EXAMPLE

7.3: Precipitation Request for Analysis and Field Information Sheet (Side 2)

SUBMISSION #

ATO_____

APIOS ORGANICS IN PRECIPITATION FIELD DATA

PGM: 02002 0401 INSTR TYPE: MICTYPE BF1 WEEK OF: 05 09 89

SISTYPE: 061 STATION NAME: DORSET

STATION ID

00000 009003011

	FIELD SAMPLE NUMBER	REMOVAL DATE DD-MM-YY	REMOVAL TIME HR-MN
1	106476	13-06-89	1200
2	106477	13-06-89	1200
3	106478	13-06-89	1200
4	106479	13-06-89	1200
5		" "	"
6		" "	"
7		" "	"
8		" "	"

	INITIAL DATE DD-MM-YY	INITIAL TIME HR-MN	PREC. AT SI	PREC. AT SN	PRE TYP	GAUGE DEPTH (mm)	GAG TYP	PRECIP TIME START HR-MN	PRECIP TIME END HR-MN	TOTAL SAMPLE VOL. (ml)	FIELD COMMENTS
1	16-05-89	1425			4	0.590	3	"	"	1230.0	K
2	16-05-89	1425			2	0.590	2	"	"	1230.0	
3	16-05-89	1425				0.590		"	"		
4	16-05-89	1425				0.590		"	"		
5	" "	"				"		"	"	"	"
6	" "	"				"		"	"	"	"
7	" "	"				"		"	"	"	"
8	" "	"				"		"	"	"	"

	STMD PRD.	COLL TYPE	SAMPLE MATRIX	QAQC CODE	BATCH NOS.	RINSE VOL(ml)	CON. GAUGE MEASURED	DEPTH SUPPLE	(MM) NOW?	DEG C	FIELD REMARKS
1	01	FI	027	A		"	3.0	"			K - PLUGGED FILTER
2	"	CT	038	A		250	3.0	"			
3	"	FI	027	C		"	3.0	"			BLANK FILTER
4	"	CT	038	C		"	3.0	"			BLANK CARTRIDGE
5						"	"	"			
6						"	"	"			
7						"	"	"			
8						"	"	"			

7.4: Air Sampler Request for Analysis and Field Information Sheet (Side 1)

APIOS REQUEST FOR ANALYSIS — WEEK OF: 14 07 89

SUBMISSION #: ATO _____

STATION ID: 00000 02500 3012

PROGRAM: 02002 0402

INSTRUMENT SERIAL NO.: HV 306

STATION NAME: DORSET INSTR TYPE: HIVOL #2

PROJ/SUBPROJ DESCRIPTION: TOXIC ORGANICS IN AIR

REMARKS: _____ TECH: J.P VARTO OPER: J.P VARTO

	FIELD SAMPLE NUMBER	SMP TYP	LAB SAMPLE NO. 1	LAB SAMPLE NO. 2	CONT S/N		DATE DD-MM-YY	TIME HH-MM
1	106480	HP			1		14 • 07 • 89	0900
2	106481	HX			1		14 • 07 • 89	0800
3							• •	"
4							• •	"
5							• •	"
6							• •	"
7							• •	"
8							• •	"

	REM1	REM2	STATIONID	DESCRIPTION	TEST GROUP	+/-
1			00000 02500 3012	FILTER ACTIVE	G • PARC	
2			"	CART. ACTIVE + ACETONE RINSE	"	
3						
4						
5						
6						
7						
8						

Use one Station ID per form.

EXAMPLE

7.5: Air Sampler Request for Analysis and Field Information Sheet (Side 2)

SUBMISSION #: ATO_____

APIOS ORGANICS IN AIR FIELD DATA

PGM: 02002 0402 INSTR TYPE: HIVOL #2 WEEK OF: 14 07 89

SITETYPE: 001 STATION NAME: DORSET

STATION ID: 00000 025C0302

	FIELD SAMPLE NUMBER	BEG. DATE-TIME DD-MM-YY	BEG. DATE-TIME HR-MN	INIT. DATE-TIME DD-MM-YY	INIT. DATE-TIME HR-MN	FIL TYP	CORRECTED FLOW VOLUME (LITRES)	FIELD COMMENTS	STND PRD	COLL TYPE	SAMPLE MATRIX	QAQC CODE
1	106480	14-07-89	0800	10-07-89	0800	1	0,23,98,8,0,0	C	22	F1	C27	A
2	106481	14-07-89	0800	10-07-89	0800	1	0,23,98,8,0,0		1	CT	OC1	A
3		" "	"	" "	"							
4		" "	"	" "	"							
5		" "	"	" "	"							
6		" "	"	" "	"							
7		" "	"	" "	"							
8		" "	"	" "	"							

	MANOMETER INITIAL	MANOMETER FINAL	ELAPSED TIME (HRS)	BATCH NOS.	FLOW CNTL. #	CALIB. ORIF. #	FIELD REMARKS
1	2.70	2.55	95.4	33	0182	8	COUNTER - 23393
2	2.70	2.55	95.4	33	0182	8	C - HIVOL FAILURE FROM
3							11/07/89 10 00 → 11/07/89 10 25
4							
5							
6							
7							
8							

REFERENCES

Bardswick, W. (1987). Technical and Operating Manual APIOS Deposition Monitoring Program (1st Revised Edition). Ontario Ministry of the Environment Report No. ARB-082-87-AQM.

Ontario Ministry of the Environment. (1990). Toxics Deposition Monitoring Program Quality Assurance Plan. Report No. ARB 060-90.